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U. S. DEPARTMENT OF AGRICULTURE.
DIVISION OF CHEMISTRY.
BULLETIN No. 16.

METHODS OF ANALYSIS

OF

Commercial Fertilizers, Feeding Stuffs, and Dairy Products

ADOPTED AT THE

FOURTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS,

AUGUST 16, 17, AND 18, 1887.

EDITED BY
CLIFFORD RICHARDSON,
SECRETARY.

WASHINGTON:
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1887.

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LETTERS OF TRANSMITTAL.

UNITED STATES DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., September 10, 1887.

SIR: I have the honor to submit for your approval the proceedings of the Fourth Annual Convention of Official Agricultural Chemists, which met, by your invitation, at the Department of Agriculture on the 16th of August, this year.

The great advantage which scientific agriculture has received from the publication by the Department of the proceedings of the two preceding conventions leads me to believe that a similar benefit will be derived from the publication of the accompanying manuscript as Bulletin No. 16, of the Division of Chemistry of this Department.

I am, respectfully,

H. W. WILEY,
Chemist.

Hon. NORMAN J. COLMAN,
Commissioner of Agriculture.

WASHINGTON, *August* 22, 1887.

DEAR SIR: I have the honor to hand you for publication an abstract of the proceedings of the Fourth Annual Convention of Official Agricultural Chemists in the United States, together with the official methods adopted for the analysis of commercial fertilizers, feeding stuffs, and dairy products during the coming year. There is also appended a list of the committees appointed by the president for 1887–'88, of the officers of the Association, and a copy of the constitution as recently amended.

I have thought it desirable to curtail the somewhat discursive discussions as involving the publication of too large a bulletin and serious delay; in fact, being unable from the slender notes at my disposal to do justice to many of the speakers, the size of the meeting having grown beyond the possibility of reporting them without stenographic assistance. The essential points of each debate and the results are, however, given, I trust, with accuracy.

The increasing demand for our proceedings shows that the wisdom of the Department of Agriculture in disseminating them has been appreciated, and is laying the foundation for uniformity among official and commercial chemists, which is of the greatest value to the agricultural and business interests of the country.

The extension of the subjects considered by the Association, will, no doubt, vastly increase its sphere of usefulness.

Very respectfully,

CLIFFORD RICHARDSON,
Secretary.

Dr. H. W. WILEY,
Chemist.

UNIFORM METHODS FOR THE ANALYSIS OF FERTILIZERS, FEEDING STUFFS, AND DAIRY PRODUCTS.

PROCEEDINGS OF THE FOURTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, HELD AT WASHINGTON, AUGUST 16, 17, *AND* 18, 1887.

MORNING SESSION, TUESDAY.

In accordance with the call of the executive committee, the Association met in the library of the Department of Agriculture at 10 o'clock, the president, Dr. E. H. Jenkins, in the chair.

The following members and "others interested in the objects of the Association" were present:

The President—Dr. E. H. Jenkins, of the Connecticut Agricultural Experiment Station.

The Vice-President—Mr. P. E. Chazal, State chemist of South Carolina.

Of the Executive Committee—Prof. M. A. Scovell, director of the Kentucky Agricultural Experiment Station and State chemist, and Dr. H. W. Wiley, chemist of the U. S. Department of Agriculture.

The Secretary—Mr. Clifford Richardson, of Washington, D. C.

Dr. William McMurtrie, of Champaign, Ill.
Dr. W. J. Gascoyne, of Richmond, Va.
Dr. S. M. Babcock, of Geneva, N. Y.
Prof. G. S. Fellows, of Agricultural College, Maryland.
Prof. William Frear, of State College, Pennsylvania.
Dr. E. A. v. Schweinitz, of Lexington, Ky.
Prof. Frank T. Shutt, of Ottawa, Canada.
Dr. Milton Whitney, of Raleigh, N. C.
Dr. Thomas Macfarlane, of Ottawa, Canada.
Dr. G. C. Caldwell, of Ithaca, N. Y.
Dr. E. H. Farrington, of New Haven, Conn.
Prof. W. C. Stubbs, of Baton Rouge, La.
Prof. W. E. Moses, of Knoxville, Tenn.
Prof. C. E. Waite, of Rolla, Mo.
Prof. N. W. Lord, of Columbus, Ohio.
Prof. J. A. Myers, of Agricultural College, Mississippi.

Prof. N. T. Lupton, of Auburn, Ala.
Prof. W. B. Rising, of Berkeley, Cal.
Prof. Robert B. Warder, of La Fayette, Ind.
Prof. Edgar Richards, of Washington, D. C.
Prof. Fred A. Holton, of Kansas City, Mo.
Prof. Charles E. Bond, of Spencerville, Md.
Prof. Charles A. Crampton, of Washington, D. C.
Prof. Arthur L. Green, of La Fayette, Ind.
Mr. A. E. Knorr, Mr. N. J. Fake, Mr. F. V. Broadbent, Mr. Felix Langfeldt, Mr. John Dugan, of the U. S. Department of Agriculture.
Mr. de Ghequier, representing the interests of the National Fertilizer Association.

Letters of regret were read from many prominent gentlemen who were unable to be present.

In calling the convention to order the president spoke as follows:

GENTLEMEN OF THE ASSOCIATION: We begin to day the fourth annual meeting of this Association. It is the eighth meeting of American agricultural chemists for the discussion of methods of analysis. In consequence of the enlargement of the scope of our discussions we hope that there will be a considerable addition to our numbers at this time, and that in consequence the Association, which has already proved itself to be of very great value both to its members and to producers and consumers of fertilizers as well, will become of even greater value in these particulars, and also in other respects, to all who are personally engaged in agricultural chemical research.

In opening the meeting I wish to call attention to one or two particulars in regard to the constitution of this Association, with the object of preventing any misapprehension on the part of new members of the Association, and which we all need to keep clearly and constantly in mind.

First, then, this is an Association of analysts in a special department of chemistry. It is primarily *agricultural* chemical methods and processes which are for discussion here, not other departments of agricultural or chemical investigation. Yet we cannot draw any very rigid line between them. For instance, the availability of the different forms of phosphoric acid is an agricultural question and a chemical question, but taken by itself would hardly concern us, and experiments on the subject would more properly be presented and discussed elsewhere than here.

But Wagner's vegetation experiments on the subject in connection with his method of determining available phosphoric acid in the laboratory, the results of which are designed to coincide with the results of vegetation experiments, would seem to bring this matter within the scope of our discussions in so far—but only so far—as they might influence us in fixing a method for determining available phosphoric acid.

It is to cover such cases as this that the second object of the Association is declared to be "to afford opportunity for the discussion of matters of interest to agricultural chemists."

Again, as to membership. The Association is for the use of all those who are engaged in agricultural investigation and experiment, or who are charged with official duties in the examination of fertilizers, foods, &c. The membership is limited to persons of these two classes.

I call attention to the fact that the wording of the second clause of our constitution is rather ambiguous, and might be so interpreted as to exclude some who belong in the classes above named. The clause in question reads:

> Analytical chemists connected with departments of agriculture, agricultural experiment stations, agricultural colleges, State boards of agriculture, and other bodies charged with official control of the materials named in section 1 shall alone be eligible, &c.

Now this may be interpreted to exclude chemists from agricultural colleges which do not exercise any *official* control over the articles named in section 1. Such an interpretation, I am sure, was not intended, and the Association may see fit to change this, either by inserting after "agricultural colleges" the words "and chemists of," or else by making the section read:

> Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with an official control of the materials named in section 1, &c.

This last form would include the chemists of the Treasury Department who are appointed under the national law regulating the sale of oleomargarine.

The provision that only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers, may, at first thought, seem rather unfair, but a little consideration will, I am sure, commend it to the judgment of our new members. The official chemists are responsible to their governing boards, to the manufacturers and the consumers alike; large financial interests are involved, and it is no more than fair that having these responsibilities, they alone should have the decision as to the methods they shall use. The necessity for this was apparent after the Cincinnati meeting of "agricultural chemists," prior to the organization of this Association, where of thirty-two members in attendance only nine at most (probably fewer) had any official connection with fertilizer control, and where a method was adopted as "official" which was promptly repudiated by official chemists.

None the less all the assistance that can be rendered by study and discussion on the part of other members of the Association and other chemists who may be present is most desirable, and in past years has been very helpful. It should be added that the National Fertilizer Exchange, representing the fertilizer manufacturers' interest, through its

secretary, Mr. D'Ghequier, has expressed itself as well satisfied with the present plan of fixing methods of fertilizer analysis.

On the conclusion of the president's address the recommendation of the executive committee that the following should be the order of business for this meeting was unanimously agreed to:

(1) Reports of committees: (*a*) On Cattle Foods; (*b*) on Dairy Products; (*c*) on Phosphoric Acid; (*d*) on Potash; (*e*) on Nitrogen.

(2) Election of officers and miscellaneous business.

In accordance therewith Dr. G. C. CALDWELL, on behalf of the Committee on Cattle Foods, submitted the following report:

REPORT OF THE COMMITTEE ON THE ANALYSIS OF CATTLE FOODS.

The Committee on the Analysis of Cattle Foods submit the following report:

Fifteen chemists connected with experiment stations or agricultural colleges consented to make analyses of samples of fodder to be prepared and sent out by the committee—some of them, however, conditionally, being uncertain whether they would have time for the work. Reports were received from ten of the number on the three samples sent out.

These samples were hay, bran, and cotton-seed meal. The first was cut up and ground in a large coffee mill, and then very carefully mixed by stirring over with the hands on a large sheet of paper. The other samples were mixed as much as seemed to be necessary to make them uniform in composition. It was assumed, however, that but little preparation of these stuffs was necessary. The portions for each chemist were taken from the dish containing the whole of the mixed sample by means of a large spoon plunged into the mass, and not by pouring.

Each analyst was requested to follow such methods of analysis as he preferred, but to give the committee full information as to the details of the methods used, either directly or by reference to generally accessible authorities.

The results returned are given in the following table, calculated to water-free substance.

[All results calculated to water-free substance.]

	Cotton-seed meal.					Wheat bran.					Hay.				
	Ash.	Protein.	Ether extract.	Fiber.	Nitrogen free, extract.	Ash.	Protein.	Ether extract.	Fiber.	Nitrogen free, extract.	Ash.	Protein.	Ether extract.	Fiber.	Nitrogen free, extract.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct*	*P. ct.*	*P. ct.*	*P ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
Babcock	7. 37	52. 20	10. 60	6. 42	23. 41	7. 17	16. 84	4. 69	11. 65	59. 65	5. 25	8. 72	3. 23	33. 49	49. 31
Caldwell	7. 33	51. 29	11. 13	...	.....	7. 20	15. 97	3. 34	10. 95	62. 54	5. 49	9. 38	3. 62	32. 22	49. 29
Frear	7. 76	44. 78	10. 95	4. 09	32. 42	7. 46	15. 87	4. 97	8. 30	63. 90	6. 31	11. 48	3. 67	26. 99	51. 55
Jordan	7. 38	52. 61	10. 90	6. 65	22. 46	7. 36	17. 43	4. 42	10. 46	60. 33	5. 73	9. 04	3. 39	29. 93	51. 91
Lupton	7. 28	50. 04	11. 87	6. 44	24. 37	6. 83	17. 00	3. 90	13. 36	58. 91	5. 64	8. 32	3. 13	33. 12	49. 79
McMurtrie	6. 89	...	8. 89	9. 20	*27. 69	.....	.....	.....	.....	..	7. 41	11. 59	1. 85	32. 69	*45. 59
Richardson	7. 44	50. 70	12. 23	5. 07	24. 55	7. 74	16. 45	6. 50	6. 91	62. 40	5. 55	8. 79	5. 36	25. 97	54. 33
Scovell	7. 44	51. 80	10. 57	3. 71	26. 48	7. 33	16. 93	2. 84	7. 39	65. 51	5. 81	8. 95	4. 07	26. 03	55. 14
Warder	7. 43	52. 73	12. 32	....	27. 51	7. 29	17. 17	5. 83	....	69. 73	5. 06	8. 56	5. 61	....	79. 38
Weber	7. 51	51. 60	10. 53	5. 95	24. 41	7. 36	17. 01	4. 85	10. 66	60. 12	5. 63	9. 41	3. 21	32. 13	49. 62

*Direct.

Total and album. N. in dry substance.

	Cotton-seed meal.		Bran.		Hay.	
	Total.	Album.	Total.	Album.	Total.	Album.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
C	8.21	8.00	2.56	2.32	1.50	0.95
B	8.34	7.9	2.70	2.10	1.39	1.21
F	7.16	7.14	2.54	2.23	1.83	1.60
M	.....	.....	.....	.....	1.68	1.68
R	.....	.....	.....	.....	1.40	1.40

In view of the general similarity in the methods of analysis employed, and, it might be added, necessarily employed, the serious divergence in the results obtained by different chemists on the same sample is rather startling and calls urgently for further work in this direction.

With regard to the ash the results agree so well, with but a very few exceptions, that this part of the subject seems to need but little further treatment, and yet even here the difference between the highest and the lowest per cent. on bran is nearly 0.9 per cent. The difference can hardly be ascribed to want of uniformity in the sample, for a peck of it could reasonably be supposed to be practically uniform in composition even as brought from the mill; so between the highest and lowest ash in hay the difference is a little over 2 per cent., or, leaving out the case in which the work was reported as being done by an advanced student in the laboratory, we have a maximum difference of 1.3 per cent. In spite of the precautions taken to secure perfect uniformity of the sample, this difference may be explained by imperfect mixing. The highest ash obtained, still making the exception above referred to of the extremely high result, was, with both cotton-seed meal and hay, with the use of an alcohol lamp instead of gas. The lowest ash in cotton-seed meal and the highest of all on hay was obtained by charring, exhausting with water, burning the residue of coal, and adding this ash to the residue left on evaporation of the aqueous extract.

With regard to the protein, when it is considered that the quantity of substance taken for analysis is necessarily small, and that even after calculating the percentage on nitrogen from the small quantity we have still to multiply by 6.25 for the final result, the results in the main agree as well as could be expected. The agreement is closest on the cotton-seed meal, excluding one report so far out as to indicate some mistake, next best on the bran, and by far the poorest on the hay.

The nitrogen was determined by some after Kjeldahl, by others by soda lime, and in one case after Ruffle; but no uniform difference in results corresponds with different methods. Apparently only in more careful preparation of the sample or more scrupulously careful work in the analyses can we hope for improvement in results on the protein.

Ether extract.—Here we have but a single operation of the simplest character, performed in the same general manner, and the product to be determined is itself weighed; and yet the results reported differ by as much as 2 per cent. on an average total of only 3 to 4 per cent. Some of the chemists used Squibbs (stronger) ether, others ordinary washed ether. Most of them appear to have used Soxhlet's self-siphoning extractor, others used an ordinary co tinuous.

One, and perhaps more, have used the same sample for the determination of moisture and of ether extract. Dr. Babcock has found that long-continued desiccation appears to lessen the solubility of the fat in ether (2nd Rep. N. Y. Exp. Sta., 1883), but this was proved only for drying at 100° for several (7) days and upward.

The duration of the time of extraction varied from a few hours to three or four days. In many cases nothing is stated as to tests for complete extraction, or as to the time of extraction.

One chemist, who reported the lowest results on bran, but on this sample only, subsequently, in a supplementary report detailing results of experiments on time of extraction, obtained by a much longer extraction, extending over some days of alternate simple contact with the ether in a self-siphoning apparatus and extraction by boiling, instead of eight hours as before, gives results on bran nearly on a par with the others, but no higher results on hay. Another, who reports much the highest results on cotton-seed meal and hay, states that from three to four days were required for complete extraction. Another (R), who gave treatment for twelve hours in a self-siphoning apparatus, reports very much the highest results on bran, and only a little less than W. on the other samples.

There appears to your committee to be ground for the hope that, by uniformity in the details of the method of carrying out this determination, much greater uniformity in results can be secured. This uniformity should refer particularly to the previous treatment of the sample, the kind of ether used, and the time of extraction. Whether the form of apparatus—that is, whether of the intermittent or continuous class—is of importance is uncertain.

The crude fiber.—Doubtless all who have taken part in this work of testing methods of analysis of feeding stuffs, expected wide divergence in results in the fiber, and certainly these expectations are not disappointed. Leaving out the results reported by one chemist on cotton-seed meal, which were not obtained by himself, but by an advanced student in his laboratory, and which differ so very much from the general average as to justify their exclusion, the results on cotton-seed meal range from 3.7 to 6.44; on bran, 2.84 to 5.83; and on hay, from 25.97 to 33.49.

The method followed was essentially the same in all cases, boiling with dilute acid of a certain strength for a certain time (thirty minutes), filtering, and washing; boiling with alkaline solution of a certain strength for the same length of time; washing with hot water, alcohol, and ether, drying, and weighing, and incineration, the loss of weight by this last operation giving the crude fiber. In general the same strength of acid and alkaline solution (1.25 per cent.) was used by all the analysts, but not in all cases, and in some instances the strength is not given. The boiling was in general, so far as stated, continued for the same length of time. In some cases the fat was previously extracted, in others not; presumably in all cases the residue finally obtained was incinerated, but it is not always so stated. There is much variation in the amount of substance taken, ranging from 1 to 4 grams, while the quantity of acid and alkali used remains the same, and generally speaking with the smaller quantity of substance taken the per cent. of fiber is smaller, but this is not uniformly the case.

No invariable difference depends upon whether the fat is previously extracted or not, so far as can be judged with reference to the few cases in which information is given on this point.

This matter of the determination of fiber in fodder has been made the subject of special investigation at more than one experiment station.

Krauch ("Bestimmung der Holzfaser und ihrer Mangel," Vers. Stat., *XXV*, 1880, p. 240) made a careful study of the residue left after treatment of the material with ether, alcohol, cold and warm water, and malt (Malzaufguss) which was then supposed to be free from fat, wax, chlorophyl, and carbhydrates. This residue was supposed to consist essentially of cellulose and lignine, with insoluble N. substance. It was prepared from three different feeding stuffs—rye (grain), graminaceous hay, and clover, and each residue was subjected to treatment with dilute acid and alkali successively and with Schulze's reagent, whose method, the author says, is the best for the preparation and determination of cellulose; but it often fails (im Sticke lässt) and is so difficult and tedious of execution as to be inapplicable to fodder analyses. Not getting constant results by this treatment of the residue with acid, alkali, &c., the author concludes that it varies so much in composition when prepared from different feeding stuffs as to indicate that the same treatment will not answer for all kinds for the

separation of cellulose from lignine, or, in other words, that the method of determining fiber must be varied somewhat according to the general character of the material analyzed. This is the special light he gives us on the matter.

Hofmeister ("Die Rohfaserbestimmung und das Holzgummi," Vers. Stat., *XXXIII.* 1886, p. 152) comes to the same conclusion even in comparing material so much alike as different kinds of oil cake, and still more when comparing bran with these. He says that in the ordinary method of determining crude fiber, matters may be left undissolved and reckoned as fiber which are not so left in the case of other stuffs.

But these investigations, while showing up some sources of inaccuracy of the ordinary determinations of fiber, give us no help in our search for a solution of the problem now in hand—how different analysts working on the same sample may get results agreeing so well together that we can have some degree of confidence in the method used.

Mr. Ladd ("Crude Fiber in Fodder Analysis," 5th Report New York Ag'l Exp., Sta., p. 374) gives the results of a comparison of five or six different methods of managing the treatment with acid and alkali, and finds a decided difference in the probable error by different methods, the method followed at that station having the lowest; but, after all, this may have been because it was a method with all of whose details he was thoroughly familiar by long practice.

Only five of the chemists made the determination of non-albuminoid nitrogen by Stutzer's method. The results are given in the following table:

	Cotton-seed meal.		Bran.		Hay.	
	Total.	Albumen.	Total.	Albumen.	Total.	Albumen.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
C	8.21	8.00	2.56	2.32	1.50	0.95
B	8.34	7.9	2.70	2.10	1.39	1.21
F	7.16	7.14	2.54	2.23	1.83	1.60
R					1.395	1.395
M					1.68	1.65

While there is in the main a fair measure of agreement between these results on the same sample, still there are differences that ought not to occur with reliable methods and good work.

RECOMMENDATION.

Ash.—None except extraction.

Ether extract.—Ether of uniform strength (Squibb's stronger), pulverization of substance to uniform standard, longer time of extraction than generally given—twelve hours at least.

Crude protein.—Kjeldahl's method and greater care in working, guarded by blank determinations to test material.

Fiber.—Exactly uniform strength of acid and alkali, time and temperature of treatment, agitation, previous extraction of the fat.

G. C. CALDWELL,
Chairman.
CLIFFORD RICHARDSON,
Of the Committee on Feeding Stuffs.

The report being open for discussion, Mr. RICHARDSON said that as one of the committee he desired to suggest digestion in bottles closed by patent rubber stoppers for the determination of crude fiber, using somewhat stronger re-agents and a water bath as a source of heat. He was of the opinion that uniformity might be more nearly reached in this way.

After considerable discussion he was authorized to make the method as described, an alternate with the original Weende, for official use during the ensuing year.*

Professor CALDWELL then drew attention to the direct determination of N. free extract as practiced by one of the Association.

Dr McMURTRIE said that the method was to invert with acid after extraction, and titrate with Fehling's solution.

Attention was then called to the differences caused on fiber determinations by the degree of pulverization of the sample, the indifferent use of potash and soda, the effect of different elevation on the boiling point, the amount of material, and whether or not previous extraction was practiced. These points were referred to the committee for consideration, which, on motion of Professor Caldwell, was completed by the addition of Dr. Babcock to fill the vacancy caused by Dr. Jordan's absence.

Some discussion then ensued in regard to the determination of fat, the quality of the ether employed, form of apparatus, time of extraction, and benefit of previous drying, all of which, the report having been recommitted, the committee were directed to consider and to report to the convention at some time during the session the methods to be recommended for the ensuing year.

By unanimous consent it was then carried that a committee should be appointed to consider the recommendations of the president in regard to changes in the constitution, to consist of four members and the president *ex-officio* as chairman, and a committee of three to wait upon the Commissioner of Agriculture and request him to address the convention and to thank him for his courtesies.

The chair appointed—

To wait upon the Commissioner, Professors McMurtrie, Stubbs, and Gascoyne;

To consider changes in the constitution, the president *ex officio*, Drs. Chazal, Myers, Gascoyne, and Stubbs.

The chairman then welcomed to the convention the visiting chemists, Professor Macfarlane, chief analyst of the Dominion, and Professor Shutt, of the experimental farm at Ottawa.

The convention then listened to the report of the committee on dairy products, Dr. Wiley announcing that, having prepared it himself, his colleagues on the committee were in no way responsible for the suggestions it contained.

REPORT OF THE COMMITTEE ON DAIRY PRODUCTS.

As chairman of the committee on "dairy products," I beg leave to submit the following report:

In the beginning of an investigation by the Association of dairy products, it appeared to me that the most important point to be considered was the possibility of agreeing upon uniform methods of analysis. The success which has attended our ef-

* The details are to be found among methods in Appendix A.

forts in this respect with commercial fertilizers has been great enough to encourage us in hoping for a like advantage in other fields of investigation.

The members of the committee being widely scattered, it was found impracticable to secure a meeting to discuss the "plan of campaign" and agree upon a line of investigation. In this condition of affairs I took the liberty, as chairman of the committee, to inaugurate a preliminary examination of methods of butter analysis and of inviting my confrères to undertake other parts of the investigation.

In order to obtain an expression of the views of the other members of the committee, namely, Drs. Armsby and Babcock, I addressed them the following letter:

"FEBRUARY 19, 1887.

"As chairman of the committee on dairy products, I write to ask your advice respecting the work we ought to do in the way of preparing something for the next meeting. I propose to send a letter to each of the active members asking him if he will make analyses of four samples of butter and butter substitutes which I will send.

"What part of the report to the convention will you prepare? Take some subject and work it out.

"Please let me hear from you soon."

To this I received the following courteous replies:

"MADISON, WIS., *February* 23, 1887.

"DEAR SIR: The committee on dairy products labors under a disadvantage as compared with the other committees in not being able to send around samples to any extent. The only way I see now is, as you suggest, for each member of the committee to take some subject, work it up as fully as may be. Our assistant chemist is now doing some work on butter analysis, and has done considerable in that line, so that I might be able to contribute something on that subject. However, I will take up anything you may desire that I think I can do anything like justice to.

"Is it your idea to include in the report any extended discussion?

"I think your proposal to send around samples of butter and butter substitutes for analysis an excellent one, and I will do my share.

"Yours truly,

"H. P. ARMSBY."

"GENEVA, N. Y., *March* 2, 1887.

"DEAR SIR: Your letter of February 19, in relation to work of the committee on dairy products, came when I was away from the station, or it would have been answered before this.

"I shall be glad to make analyses of any samples of butter or butter substitutes which you may send.

"I shall be working most of the summer upon milk and butter, and would prefer to prepare something in this line for the report. If you have any choice as to what points I shall devote especial attention to, please let me know and I will endeavor to attend to it.

"Very truly yours,

"S. M. BABCOCK."

On the receipt of these letters I sent the following circular letter to members of the association and others whom I thought would be interested in the matter:

"WASHINGTON, D. C., *February* 21, 1887.

"DEAR SIR: As chairman of the committee of the A. O. A. C. on dairy products, I am ready to send out some samples of butters for comparative examination. The distribution of samples of milk is of course impossible, and I propose, therefore, to send three or four samples of butter and butter substitutes. I do not think a larger

number of samples would be advisable on account of the great amount of analytical work which they would require.

"Please let me know if you wish a set of four samples sent you. If so, they will be forwarded within a few days after receiving your reply.

"Respectfully,

"H. W. WILEY,

"Chairman Committee A. O. A. C. on Dairy Products."

The following chemists expressed a willingness to undertake the analyses, viz:

Name.	Address.
Lupton, Prof. N. T.	Auburn, Ala.
Johnson, Prof. S. W	New Haven, Conn.
White, Prof. H. C	Atlanta, Ga.
McMurtrie, Prof. Wm	Champaign, Ill.
Huston, Prof. H. A	Lafayette, Ind.
Jordan, Prof. W. H	Orono, Me.
Kedzie, Prof. R. C	Agricultural College, Mich.
Babcock, Dr. S M	New York Agricultural Experiment Station, Geneva, N. Y.
Caldwell, Prof. G. C	Ithaca, N. Y.
Stillwell, Chas. M	New York, N. Y.
Frear, Dr. Wm	State College, Pa.
Moses, Prof. W. E	Knoxville, Tenn.
Traphagen, Mr. F. W	Staunton, Va.
Cooke, Prof. W. W	Burlington, Vt.
Armsby, Dr. H. P	Madison, Wis.

EXAMINATION OF BUTTER.

It being impossible to secure analyses in the way of milk samples, I prepared samples of butter and butter substitutes and sent them to the above addresses.

The samples were purchased in the open market at Washington and consisted of the following items:

No. 1.—Oleomargarine, at 20 cents per pound.

No. 2.—Butterine, at 20 cents per pound.

No. 3.—Butter, at 20 cents per pound.

No. 4.—Philadelphia print butter, at 60 cents per pound.

The samples were carefully packed in tin boxes and sent by mail. Accompanying these samples was the following letter:

"WASHINGTON, D. C., *April* 12, 1887.

"SIR: On behalf of the committee on dairy products I send you by mail this day four samples of butter and butter substitutes, and beg you to submit them to an examination.

"The examination should, if possible, comprise the following points:

"(1) A microscopic examination of a portion of the sample with polarized light and a selenite plate.

"(2) A further microscopic examination of the sample after boiling for a few moments in a test tube until the water is expelled and subsequently cooling slowly and allowing to stand for 24-48 hours.

"(3) A determination of the Sp. Gr. of the filtered and dried sample.

"(4) A determination of the melting point of the filtered fat.

"(5) A determination of the volatile acid in the filtered fat according to the principle of Reichert.

"(6) A determination of the insoluble acids in the filtered fats after washing out all that is soluble.

"(7) A determination of the saponification equivalent according to the method of Koettstorfer.

"(8) A determination of the percentage of moisture.

"(9) A determination of the percentage of salt.

"(10) A determination of the per cent. of curd.

"Since butters are apt to change in character on long keeping, I desire to urge upon you the necessity of finishing this analytical work promptly.

"When the work is completed please send the results to me for tabulation according to the usage of the association.

"Any modification or change in the methods of analysis employed, from those in general use, should be noted.

"I also desire to have all analysts engaged in this work to send me at an early date any notes or suggestions which may seem of interest for the report to be made at the next meeting of the association.

"Respectfully,

"H. W. WILEY,

"*Chairman Committee on Dairy Products A. O. A. C.*"

RESULTS OF THE COMPARATIVE ANALYSIS OF BUTTER SAMPLES.

I give below in tabular form the results of the analyses of butter, comprising all the returns that have been received from the samples sent.

Following the custom of the association, I have designated the different returns by the letters of the alphabet.

In no case has any one analyst extended his examinations to all the points suggested in the circular. The results of analyst A, however, embrace all the points mentioned but one. This omission is compensated for, however, by a study of the viscosity of the samples.

Analyst.	Polarized light and selenite plate.	Polarized light and crystallized fat.	Specific gravity.	Melting point.	Volatile acids, Reichert's method, No. cc. $\frac{n}{10}$ alkali.	Soluble acids.	Insoluble acids.	Saponification equivalent.	Water.	Salt.	Curd.
Sample No. 1.				°	*P. ct.*	*P. ct.*	*P. ct.*		*P. ct.*	*P. ct.*	*P. ct.*
A	Colors variegated.	Cross well defined.	.905	32.0	.60	.....	93.78	287.2	7.72	2.12	.47
B	.....	.....	.9079	32.2	.38	.....	91.26	283.0	7.18	2.18	1.11
C	.....	.....	.8601	.....	.60	.....	.....	.....	6.30	.....	.....
D	.....	.....	.9063	33.6*	.....	.....	.....	280.8	7.12	2.11	1.48
E	.....	.....	.9063	.....	.....	.22	95.25	.....	6.25	2.11	1.00
F	.....	.....	.....	.....	2.24	.....	95.17	275.8	6.54	1.82	.60
G	.....	.....	.....	.....	.31	.21	94 61	.....	.....	.....	.....
H	Variegated field.	.....	.9060	31.8	0.40	.....	.....	281.0	6.49	1.99	.34
Sample No. 2.											
A	Colors variegated.	Large cross	.9041	39.5	0.90	.....	94.45	288.5	7.50	2.11	.27
B	.....	.....	.9071	33.3	.29	1.19	93.40	287.0	8.06	2.21	.93
C	.....	.....	.8589	.....	.50	.....	.....	.....	6.60	.....	.....
D	.....	.....	.9056	41.5	.....	.....	.....	283.1	7.63	2.11	1.47
E	.....	.....	.9038	.....	3.90	0.10	95.90	.....	6.73	1.88	2.34
F	.....	.....	.....	.....	.....	..	96.13	263.0	6.89	1.99	.....
G	.....	.....	.....	.....	.30	.54	94.10	.....	.....	.....	.....
H	Variegated field.	.....	.9046	40.1	0.40	.....	.....	288.7	6.64	2.11	.58
Sample No. 3.											
A	Color nearly uniform.	Small cross like No. 1.	.9087	35.0	12.30	.....	85.44	250.4	11.06	5.11	.65
B	.....	.....	.9121	28.3	12.35	5.56	84.29	249.0	10.25	4.95	1.44
C	.....	.....	.8623	.....	12.70	.....	.....	.....	10.30	.....	.....
D	.....	.....	.9088	36.0	.....	.....	.....	247.7	10.06	4.45	3.70
E	.....	.....	.9086	.....	.....	4.95	89.03	.....	10.51	3.86	3.41
F	.....	.....	.....	.....	13.43	.....	90.09	241.2	10.37	4.53	.41
G	.....	.....	.....	.....	13.13	4.48	87.21	.....	.....	.....	.....
H	Uniform tint.	.....	.9091	34.5	13.06	.....	.....	244.0	8.97	4.56	.78

* At 100° C.

Results of the comparative analysis of butter samples.—Continued.

Analyst.	Polarized light and selenite plate.	Polarized light and crystallized fat.	Specific gravity.	Melting point.	Volatile acids, Reichert's method, No. cc. $\frac{1}{10}$ alkali.	Soluble acids.	Insoluble acids.	Saponification equivalent.	Water.	Salt.	Curd.
Sample No. 4.				°	*P. ct.*	*P. ct.*	*P. ct.*		*P. ct.*	*P. ct.*	*P. ct.*
A	Color uniform.	Crystals, large cross.	.9114	34.4	14.4	.....	87.06	249.0	10.95	1.00	.65
B	.....	.....	.9137	26.6	14.31	5.75	83.37	250.0	11.36	.87	1.34
C	.....	.....	.8645	...	14.60	.....	.....	... ..	9.80	.....	.....
D	.....	.....	.9105	35.4	.....	....	.....	244.5	10.86	.87	1.79
E	.....	....	.9110	.....	.....	5.81	86.94	.....	8.21	.79	1.32
F	.....	.....	.....	.....	15.75	.....	88.60	230.9	11.03	.86	.....
G	.....	.....	.....	.....	15.90	5.73	87.43	.....	.....	.....	.....
H	Uniform tint.	.....	.9109	33.0	15.39	.....	.....	248.0	8.44	.79	.64

SUPEREROGATORY ANALYSES.

In two instances, viz, A and H, the analyses were pushed further than was asked for in the circular letter already quoted.

The viscosity of the soap solutions made from the fats of the samples was determined by Analyst A.

Following are his results:

Sample.	
No. 1	Too viscous to test.
No. 2	Do.
No. 3	Viscosity = 173.
No. 4	Viscosity = 109.

The determination in each case was made with 15 grams filtered fat and 10 grams KOH, the volume being made up to 500 cc. with water.

The soaps from Samples 1 and 2 gave solutions too viscous to be determined by the apparatus as arranged. This means a viscosity exceeding 1,000 on the scale adopted.

IODINE ABSORPTION.

The iodine absorption of the several samples was determined by Analyst H. Following are the percentages of iodine absorbed.

The determinations were in quadruplicate.

Number of determination.	Iodine absorbed.			
	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
No. 1	63.23	58.28	37.81	21.15
No. 2	63.28	58.57	37.67	20.98
No. 3	64.01	58.54	37.79	20.89
No. 4	63.93	58.22	37.59	20.85
Mean	63.61	58.40	37.72	20.97

METHODS OF ANALYSIS EMPLOYED.

In the absence of any method of examination agreed upon by the members of the association each analyst was left free to follow that process which seemed to him best.

Complying with the request of the circular letter, however, a brief sketch of the methods employed accompanied most of the returns received.

DESCRIPTION OF METHODS OF ANALYSIS.

Following is a recapitulation of the several schemes of analysis that were pursued:

Analyst A.—The specific gravity was determined at 40° C. by weight in a specific-gravity bottle. The melting points were found by Wiley's method. The volatile and insoluble acids and the saponification equivalent were determined in the usual manner. The moisture, salt, and curd were found by methods described in New York Agricultural Experiment Station report for 1886, the salt being considered the same as ash, and the curd calculated from nitrogen.

Analyst B.—Specific gravity of filtered fat determined by a 10 cc. specific-gravity bottle at 37°.7 C., according to directions given by Blyth (see Blyth, Foods Composition and Analysis, London, 1882, p. 295); duplicate determinations made.

"Melting point of butter fat determined by a small glass bulb weighted with mercury (*loc. cit.*, p. 294). Weight of bulb, 3.7806 grams. Temperature noted at the point when the bulb sank just below the surface of the fat in the test tube (diameter of test tube 1 inch).

"Reichert's method: As nearly 2.5 grams of the filtered fat as possible were weighed into a 4-ounce flask while in a fluid state. Twenty cubic centimeters of alcohol (80 per cent.) and about 1 gram of solid KOH were added; the flask was heated on water bath till the air sucked out of it no longer smelled of alcohol; 50 cc. of water were then added, and when the soap was wholly dissolved, 20 cc. of sulphuric acid (1–10). Two pieces of pumice stone attached to short platinum spirals were used to prevent bumping in distilling. The distillate was passed through a small wet filter into a 50 cc. flask; the first 20 cc. of distillate being poured back if found necessary, and was titrated with decinormal soda solution, using phenolpthalein as an indicator; duplicate determinations being made, and 0.07 cc. of NaOH sol., being required by blank test, always deducted from results.

"Hehner's method: Carried on as directed by Blyth (*loc. cit.* 298); to about 4 grams of fat weighed out in a 4-ounce flask, 50 cc. of semi-normal alcoholic solution of potash were added; flask covered with a small watch glass, and heated slowly till all fat saponified; watch glass then removed and alcohol evaporated; when wholly free from alcohol, soap dissolved in about 50 cc. of water, solution transferred to a 500 cc. flask; soap decomposed with semi-normal H^2SO_4 and insoluble fatty acids washed as directed. Only single determinations made."

As will be seen, results are all lower than usually given, both in case of samples of butter and of butterine. Analysis of samples made in spring, 1886, with other solutions showed similar results, as will be seen from the following table:

Sation No.	Insoluble acids.	Soluble acids.	Sum.	Pure butter.
	Per cent.	*Per cent.*	*Pr. ct.*	*Per cent.*
1	86.09	4.88	90.97	100
3	86.01	5.27	91.22	100
8	96.02	.75	96.77	(*)
14	90.52	.00	90.52	(†)
15	85.52	5.92	91.42	100
20	91.08	.02	91.10	(†)
23	84.27	5.93	90.20	100
24	93.23	1.03	94.26	(*)
30	86.55	4.90	91.45	80
32	86.33	5.11	91.44	100

* Oleo oil. † Oleomargarine.

The writer is unable to explain these low percentages; whether the mistake is in the manner of proceeding or in the method. A series of experiments with No. 3, a sample of pure creamery butter, clearly pointed out that evaporation of *all* alcohol defore dissolving the soap is essential. The following were the results:

No.	Alcohol not evaporated.			Alcohol evaporated.		
	Insoluble.	Soluble.	Total.	Insoluble.	Soluble.	Total.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	84.36	4.95	89.31	.....	5.41	.....
2	83.42	4.79	88.20	85.95	5.06	91.01
3	85.02	4.93	89.95	86.07	5.35	91.42
Average	84.20	4.89	89.15	86.01	5.27	91.22

"Koettstorfer's method, carried on as directed by the author (see Blyth, Foods, p. 296); results given are average figures of two or more determinations.

"Water, determined by drying in copper oven for one hour at 100° C. in a current of carbonic acid. the details of the process will be discussed in a future publication.

"Ash, determined by Dr. S. M. Babcock's method (see fifth annual report of New York Agricultural Experiment Station, p. 335), about 3 grams of butter taken for determination. Duplicate determinations made; duplicates agreed within .04 of 1 per cent., the following being the results: 2.14 and 2.18; 2.20 and 2.21; and 4.94 and 4.95; .85 and .88.

"Caseine (curd), determined by Kjeldahl's process, the fat of the butter being previously dissolved out by ether. About 2 grams of butter taken for the determination."

Analyst C.—Volatile fatty acids, expressed in cubic centimeters of $\frac{1}{10}$ normal NaOH solution, determined by Reichert's method.

"Specific gravity of filtered fat by Westphal's balance at 100°C., as described in report Connecticut Agricultural Experiment Station, 1886.

"Water determined by heating 2 grams at 100° and then at 110°C., in flat platinum dish two to three hours."

Analyst D.—The moisture, curd ash, and total fat by direct weighing were estimated as follows:

"Into a small beaker of known weight was placed a short-necked funnel fitted with a plaited double filter. A second weighing was made. The sample was now thoroughly mixed and enough added to the funnel to weigh exactly 10 grams. The beaker funnel and contents were placed in an air bath at 105° for about one and one-half hours. By this manipulation the curd and salt are separated from the fat, and the water is driven off, all in one operation. I found that a single filter would not always retain the curd and salt completely. The filter was exhausted with ether into a second weighed beaker, the ether evaporated, and the weight of this fatty residue added to the weight of the first filtrate. The filter was ignited for ash. The specific gravity was taken in ordinary specific gravity bottles holding about 25 grams and fitted with perforated ground-glass stoppers. The body of the bottle was immersed in a water bath and the temperature gradually raised to 40°. This rise of temperature proceeded slowly. For Koettstorfer's equivalent, Allen was followed."

Analyst E.—Does not report methods employed. Samples 1 and 2 are reported to be artificially colored. No. 3 is reported to be adulterated with 15 per cent. foreign fat; allowing 88 per cent. to be the average of insoluble acids.

Analyst F.—The curd was determined by first exhausting with ether, drying and igniting the residue.

"Salt by the volumetric method with $KCrO_4$ as indicator, as directed in Bulletin No. 13, part 1, pages 73 and 74."

Analyst G.—The determination of volatile acid, soluble and insoluble acids, was made according to the methods described in Bulletin No. 13, part 1, pages 57, 66, and 67.

Analyst H.—The determinations were made by the methods described in Bulletin No. 13, part 1, viz: Specific gravity, pages 40 and 41; volatile acids, pages 57 and 58; melting point, page 45 *et seq.*; water, by drying on sand for two hours at 105°; salt, pages 73 to 74; curd, page 74; iodine absorption, pages 63 to 64.

PHYSICAL CHARACTERISTICS.

The examination of the butter samples with the microscope was only made by two of the analysts, viz: A and H. Their results are entirely concordant, save that in No. 3. A reports a slight appearance of prismatic colors in the field of vision with the selenite plate which was not observed by H.

This agreement lends emphasis to the opinion expressed in Bulletin No. 13, part 1, page 39. The boiling of the suspected fats and their subsequent examination, after crystallization, with polarized light, appear to be of no value whatever in qualitative separation of butters.

SPECIFIC GRAVITY.

The agreement in specific gravity determinations is reasonably encouraging.

With the exception of the results of C, which were made by a separate method, the following points are noticed. A D and H were determined in picnometers at 40°.

Sample.	Results.		
	A.	D.	H.
No. 1	.9050	.9063	.9060
No. 2	.9040	.9056	.9046
No. 3	.9087	.9086	.9091
No. 4	.9114	.9105	.9109

No other methods were employed with more than one sample. At least in the absence of any report irrespective of E the above remark is true.

The results at 40°C. in picnometer are so concordant that it will not be difficult to secure the adoption of this method by the Association.

MELTING POINT.

Reports A. and H. follow the method of the author. (Bul. No. 13, part 1, pp. 45 *et seq.*) Following are the data:

Sample.	Melting point.	
	A.	H.
	° *C.*	° *C.*
No. 1	32.0	31.8
No. 2	39.5	40.1
No. 3	35.0	34.5
No. 4	34.4	33.0

No other method was used by more than one analyst, and hence a like comparison cannot be made.

The results of the other methods differ so widely from each other and from the nearly agreeing results of the author's process as to show the small value which they possess.

The agreement in the comparison given is so close as to excite a hope that the method, when strictly followed in all its details, will afford results which are valuable. This value is especially noticeable in sample No. 2, where the melting point alone indicates a "neutral lard."

CHEMICAL CHARACTERS.

Volatile acids.—The value of the distillation method for sorting butter is again manifested in the results under consideration.

The process having been essentially the same with all the analysts, the whole number of results may be directly compared.

The most noticeable feature of this comparison are the peculiar results obtained by F, results which could only have arisen through some error of manipulation, probably due to some of the sulphuric acid being carried over during the process of distillation.

These errors of F, however, are confined to samples Nos. 1 and 2 and disappear in 3 and 4. This circumstance renders their occurrence the more inexplicable.

The results for samples 3 and 4 are decidedly satisfactory. When it is considered that a systematic chemical examination of butter is almost a new branch of chemical work in this country, the agreement of the results must be considered a cause of congratulation.

The two samples of butter, viz., 3 and 4, happily represent very nearly the mean and maximum of decinormal alkali consumed by the distillate obtained by Reichert's method. It is easy to see, however, that with a butter like No. 4, requiring nearly 16 cc. N. $\frac{1}{10}$ alkali for the distillate from 25 grams, a considerable adulteration could be produced without exciting suspicion, if examined by Reichert's method alone.

Thus 1 gram of No. 1 would give .1 cc. and 4 grams No. 4 12.8 cc., in all 12.9 cc., which would be declared pure butter if examined by distillation alone. Such a mixture, however, viewed by polarized light through selenite would show prismatic colors and crystals of the adulterant.

Its specific gravity would also be 1 gram No. 1 = .9050; 4 grams No. 4 = 3.6436; 5 grams = 4.5486; specific gravity of mixture = .9097.

This number is not convincingly low, but, taken with the variegated field of the microscope, would be good circumstantial evidence. Continuing the comparison further in respect of the melting-point, it is seen that the mixture of 1 part of No. 1 with 4 parts of No. 4 would not affect the melting-point in a noticeable manner. But the case is quite different if mixed with No. 2.

Here we have the following data: 1 part No. 2, 40°; 4 parts No. 4, 132°; 5 parts 172°, and the melting-point of the mixture would be 34.4°. This is not above the maximum for a pure butter, but the average for pure fresh butter is about 33.8°. Thus another circumstantial proof would be brought into court.

Nevertheless, the distillation test is the best single chemical operation to establish the purity of a butter.

Insoluble acids.—The determination of insoluble acids appears to have been made essentially by the same process, and the results show that it requires special training on the part of analysts to secure uniform results.

I may say, not having made them myself, that the results reported by analyst G have behind them more than two years of quite constant practice of the method, and I am inclined, without disparaging the data of the other analysts, to take these as a basis of comparison.

The results for the several samples are as follows:

No.	Per cent.
1	94.61
2	94.10
3	87.21
4	87.43

I will say of the above results, that if they are not correct, then neither long practice nor a high degree of analytical skill can secure accuracy.

You will only have to glance at the tabulated results to see that in the case of these acids it is necessary to carefully watch the minute details of the manipulation. These details are carefully set forth in the scheme of analysis submitted herewith for the consideration of the Association.

Saponification equivalent.—The general agreement is good and shows that with careful manipulation the results are strictly comparable. The method recommends itself by the ease and rapidity of manipulation, as will be seen in the appended scheme of analysis.

Water.—The variations in the per cent. of water obtained are somewhat surprising and illustrate again the necessity of the adoption of some uniform method by the Association. The water, however, is not uniformly distributed in the butter, and hence variations from this cause must be expected.

Salt.—The agreement here is also encouraging. The salt was estimated as ash by Babcock's method. (5 Ann. Report N. Y. Ag. Exp. Sta., p. 335) by A and B.

Analyst.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.
A	2.12	2.11	5.11	1.00
B	2.18	2.21	4.95	.87

It would be unreasonable to demand a better agreement than the above. The method is shown to be most excellent.

By analysts F and H the salt was determined volumetrically by the author's method.

Results.

Analyst.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.
F	1.82	1.99	4.53	.86
H	1.99	2.11	4.56	.79

In these two sets of analyses we have also a most satisfactory agreement. The per cents are slightly less than in the first set, for this method determines nothing but NaCl. It would be well to combine it with Babcock's method and thus determine both ash and NaCl. If the ash should show any notable increase over the salt, it would indicate the addition of borax, sodium carbonate, or some other mineral substance to the butter.

Curd.—A and B determined curd by conversion into ammonia by Babcock's method. Following are the results obtained:

Analyst.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.
A	.47	.27	.65	.65
B	1.11	.93	1.44	1.34

B's results are uniformly higher than A's. The method, therefore, needs to be followed in uniform detail.

In a general review of the analytical data may be seen the fact that, while there is a near agreement in many instances, yet much is to be done before the work of the various members of our Association can be considered strictly comparable.

In view of securing greater uniformity in this respect, I beg leave to submit for the consideration of the members of the Association, the following outlines of methods for the coming year:

[The recommendations of Dr. Wiley, on behalf of the committee, were adopted with a few changes, which are mentioned in the following discussion and appear among the official methods in Appendix A.]

Dr. Wiley then continued his report in relation to milk:

It is not possible to prepare and send samples of milk for comparative analysis as was done with butter samples. Therefore, I am not able to present to the association data which illustrate the merits or demerits of the different processes of analysis.

It seems best, therefore, that I should submit for your consideration an outline of analytical methods which experience has shown to be reliable. With such modifications as may be made in this "committee of the whole," I feel sure that by following such a uniform method uniform results will be obtained.

Within a short time the methods of milk analysis have undergone a radical change and in scarcely any other branch of agricultural chemical analysis would schemes of examination prepared even two years ago, and at the present time show such marked differences.

The old methods of gravimetric determinations of fat in milk are almost entirely obsolete, while, of the volumetric methods, only the modified form of Soxhlet's process and the determination by the lactocrite have met with common acceptation.

In this connection also I must not fail to mention the method of Morse, in which anhydrous copper-sulphate is used for drying the milk, and the fat, after extracting with petroleum, is estimated by saponification. In preparing the following scheme for the examination of milk I have thought it best not to recommend Soxhlet's method for two reasons, one of which is that I have shown that its results, as computed by the table, are slightly too low, and especially because all the members of the association may not be provided with the apparatus.

H. W. WILEY, *Chairman.*

The recommendations as amended were adopted and appear among the official methods on the Appendix.

On the conclusion of the report the convention adjourned to meet at 2 p. m.

TUESDAY AFTERNOON.

The convention was called to order at 2 p. m., and, the discussion of the report of the committee on dairy products being in order, agreed by unanimous consent to listen instead to the report of methods from the committee on cattle foods as agreed upon after recommitment.

The committee reported for use during the ensuing year the methods given in the Appendix, and their report was adopted.

On motion of Dr. McMURTREE it was voted: That the committee on cattle foods be directed to collect such information on the subject of the determination of water as can be obtained, and recommend some method at the next meeting for adoption.

The convention then entered on the discussion of the report of the committee on dairy products, and the reading of papers being in order the following were presented:

AVERAGES OF ANALYSES OF AMERICAN BUTTERS, BY EDGAR RICHARDS, CHEMIST TO THE UNITED STATES BUREAU OF INTERNAL REVENUE.

In connection with the report of the committee on dairy products the following tables giving the analyses of samples of genuine butter and of some butter substitutes may prove of interest.

The methods pursued in the analyses were those recommended by the chairman of the committee, in his report just read, and which have been followed by the division of chemistry of the Department of Agriculture since 1883.

A table showing the analysis of various fats, compiled from Allen's Commercial Organic Analysis, 2d edit., vol. 2, is presented for comparison.

TABLE I.—*Analyses of genuine butter, American.*

[Analysts Crampton and Richards, Department of Agriculture, 1883–'86.]

	Water.	Casein.	Salt.	Specific gravity at 40° C.	Soluble acids.	Insoluble acids.	Saponification equivalent.	Vol. $\frac{N}{10}$ NaOH for 2.5 grams.
Fifty-two samples:	*Per cent*	*Per cent.*	*Pr. ct.*		*Per cent*	*Per cent*		
Average	10.49	.599	3.27	.91128	4.77	87.79	247.6	13.50
Highest	17.44	1.230	7.10	.91250	6.79	89.55	268.5	15.60
Lowest	4.44	.263	1.08	.90995	3.00	86.43	236.5	12.30
Fresh butter, 26 samples:								
Average	11.42	.622	2.48	.91105	4.81	87.99	248.6	13.90
Highest	17.44	.938	6.15	.91235	6.79	89.26	268.5	15.30
Lowest	8.14	.263	1.08	.91030	3.00	86.43	239.8	12.90
Tub butter, 26 samples:								
Average	9.57	.602	4.08	.91151	4.73	87.78	246.7	13.40
Highest	13.67	1.230	7.10	.91250	5.94	89.55	254.2	15.60
Lowest	4.44	.263	1.81	.90995	3.47	86.60	236.5	12.30
Individual cows:								
Jersey cow, value 2d.	9.98	.660	1.64	.91102	6.79	86.72	239.8	.....
Do	12.54	.622	1.64	.91089	4.52	88.02	247.1	.....
Jersey cow, Washington, D. C	11.89	.263	2.61	.91073	4.69	87.71	244.9	14.10
Celebrated dairies:								
Hampton, Baltimore, Md	14.06	.507	2.78	.91149	4.97	88.46	247.3	.....
Darlington, Darlington, Pa.	11.67	.455	1.42	.91049	4.21	89.26	252.8	.....
Do	11 46	.831	1.48	.91165	5.49	87.50	250.1	13.10

TABLE II.—*Analyses of butter substitutes.*

[Analysts Crampton and Richards, Department of Agriculture, 1883–'86.]

	Water.	Casein.	Salt.	Specific gravity at 40° C.	Soluble acids.	Insoluble acids.	Saponification equivalent.	Vol. $\frac{N}{10}$ NaOH for 2.5 grams.
Suspected butter, 13 samples:	*Per. ct.*	*Per cent.*	*Pr. ct.*		*Per cent*	*Per cent.*		
Average	10.55	.624	2.96	.90948	3.94	88.66	253.1	12.30
Highest	12.92	.963	5.40	.90987	4.84	89.89	260.1	13.10
Lowest	7.45	.297	1.50	.90862	3.02	87 60	249.4	11.60
Oleomargarine	9.32	.087	4.03	.90360	0.00	94.80	283.9	.....
Do	5.07	.172	3.31	.90488	0.20	93.42	282.5	.....
Do	10.28	.305	2.81	.90510	0.56	93.65	280.7	.....
Do	9.34	.350	3.64	.90490	0.12	93.59	274.0	0.70
Average	8.50	.229	3.45	.90462	0.22	93.87	280.3	.....
Butterine	11.69	.306	2.39	.90569	1.16	92.90	274.8	4.30
Do	14 45	.875	2.42	.90561	0.09	93.72	281.1	1.90
Lard, leaf	0.00	.000	0.00	.90460	0.00	95.40	284.7	.....
Do	0.00	.088	0.00	.90538	0.41	92.59	294.3	0.20
Lard, neutral	7.42	.....	0.40	.90369	0.20	90.00	270.5	0.30
Beef, suet	0.00	.....	0.00	.89897	0.00	94.80	280.0	.....
Do	0.00	.....	0.00	.90158	0.22	92.59	296.6	0.10
Oleo fat	14.23	.....	0.97	.90287	0.10	93.35	286.2	0.20

TABLE III.—*Average analysis of various fats.*

[By A. H. Allen.]

	Water at 15.5° C.=1.				Water at 37° C.—1.		KOH for saponification.
	Specific gravity.	At—	Specific gravity.	At—	Specific gravity.	At—	
		°		°		°	*Per cent.*
Butter fat	.9041	40	.8677	99	.9094 to .9140	37.8	22.15 to 23.24
	.926 to .929	15.5	.867 to .870	99			
Butterine	.8982	40	.8592	98	.9014 to .9060	37.8	19.35 to 19.65
	.915	15.5	.8585 to .8630	98			
Lard	.8985	40	.8608	98	.9038 to .9050	37.8	19.20 to 19.65
Lard oil	.915 to .916	15.5					19.10 to 19.60
Tallow	.8950	50	.8626	98	.9028 to .9037	37.8	19.32 to 19.80
Tallow oil	.916	15.5					
Drippings					.904 to .907	37.8	19.65 to 19.70
Cotton-seed oil	.925	15.5	.8725	98			19.10 to 19.66
	.922 to .930	15 to 15.5					
Cotton-seed stearine			.866	98	.911 to .912	37.8	
Olive oil	.914 to .917	15 to 15.5					18.93 to 19.60

	Koettstorfer's equivalent	Vol. $\frac{N}{10}$KOH per 2.5 grams.	Acids insoluble.	Acids soluble.	Solidifying point.
			Per cent.	*Per cent.*	°
Butter fat	241 to 253	12.5 to 15.2	86.5 to 89.0	4.5 to 7.0	20 to 30
Butterine	285 to 290	0.2 to 1.6			18 to 38
Lard	286 to 292				27 to 44
Lard oil	285 to 293				−4 to +10
Tallow	283 to 289				33 to 48
Tallow oil					0 to +6
Drippings	284 to 285				
Cotton-seed oil	285 to 293	0.3			1 to 4
Cotton-seed stearine	285 to 294				
Olive oil	284 to 296				+4 to −6

TABLE III.—*Average analysis of various fats*—Continued.

	Melting-point.	Br. absorption, Mills.	Iodine absorption.		
			$\frac{1}{50}$ Br. absorption.	Hübl.	Other observers.
	°	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Butter fat	29 to 35	24.5 to 27.9	38.9 to 44.4	26.0 to 35.1	19.5 to 38.0
Butterine	34 to 40	36.3 to 39.7	57.7 to 63.1	55.3	50.0
Lard	28 to 45	37.3	59.3	59.0	61.9
Lard oil					
Tallow	36 to 49			40.0	
Tallow oil					
Drippings					
Cotton-seed oil		50.0	79.5	105 to 108	106 to 109
Cotton-seed stearine	32				
Olive oil		54.0 to 60.6	85.9 to 96.4	81.6 to 84.5	83.0

VARIATIONS IN THE COMPOSITION OF BUTTER, BY DR. S. M. BABCOCK, OF THE NEW YORK AGRICULTURAL EXPERIMENT STATION.

I had the good fortune to obtain from the dairy show held in New York in May, 1887, twenty-six samples of butter for analysis. Fourteen of these were from the exhibition butters, and included several of the prize packages; the others were butters made at the fair from the milk of single cows entered for the sweepstakes butter prize. Nearly all were from thoroughbred stock, the Jerseys and Holsteins predominating.

These samples were tested by several of the methods generally employed by chemists for the detection of adulterations, viz: The volatile fatty acids used by Reichert's test, the saturation equivalent by Koettstorfer's method, the iodine number, as proposed by Hübl, the melting-points, and the viscosity of aqueous solutions of the potash soaps. Reichert's, Hübl's, and Koettstorfer's tests were made in the usual way. The melting-points were determined by Wiley's method. The details of the viscosity test are as follows: To 15 grams of the filtered fat, in a beaker of about 500 cc. capacity, are added 10 cc. of a strong solution of potassium hydrate (1 cc.=0.5 grams KOH) and 10 cc. of alcohol (95 per cent). The beaker is placed upon a water bath and heated with occasional shaking till the fat is saponified and the alcohol driven off. About 350 cc. of water are then added and heated till the soap is completely dissolved, after which 10 cc. more of the potassium hydrate solution are added. When the precipitate which forms is dissolved, the solution is transferred to a 500 cc. flask and made up to the mark with water. After it has cooled to 20° C. the volume is adjusted to 500 cc., and the viscosity determined by any reliable viscometer. The apparatus which I have used is described in the report of the New York Agricultural Experiment Station for 1886, also in the Journal of Analytical Chemistry, April, 1887. This apparatus has worked satisfactorily, the slightest difference in viscosity being shown. This sensitiveness is very desirable for determining variations in the com-

position of butter fats, but for the detection of adulteration is not necessary. Nearly all cases of commercial adulteration will be shown by simple inspection, as their soap solutions are very viscous. In most cases where doubt exists, an observation of the time required for a given volume of the solution to run from a large pipette with small jet will be sufficient to show whether the sample is butter or a substitute. The viscosities given in my work are all expressed by the number of grams of cane sugar which, dissolved in water and made up to one liter, make a solution of the same viscosity as the liquid under examination.

The results of all of these tests are presented in the accompanying table, the butter being arranged according to breed of cow so far as this is known.

Kind of butter.	Reichert's test n-10 NaOH cc.	Hübl's test iodine number.	Koettstorfer's test mgr. KOH for 1 gm. of fat.	Melting-points °C.	Viscosity of soap solutions.
Jersey butters:					
No. 1	16.1	34.5	227.5	32.7	103
No. 2	14.1	30.5	227.1	35.3	54
No. 3	13.2	35.3	229.8	33.7	71
No. 4*	14.1	32.6	231.1	33.8	81
No. 5*	11.5	35.6	223.1	33.2	97
No. 6*	13.2	25.9	232.5	35.4	50
No. 7*	14.4	23.8	232 2	34.0	65
Average	13.8	31.2	229.0	34.0	74
Holstein butters:					
No. 8	16.1	41.4	224.0	34.6	297
No. 9	12.5	41.0	220.5	34.9	256
No. 10*	14.4	44.7	224.3	32.1	461
No. 11*	14.1	40.1	226.8	32.4	179
No. 12*	13.9	42.2	227.6	30.5	187
No. 13*	12.1	34.6	222.7	36.9	166
No. 14*	16.0	36.1	229.4	32.4	112
Average	14.1	40.0	225.0	33.4	237
Guernsey butter:					
No. 15	15.6	36.9	227.2	33.5	153
No. 16*	14.3	33.9	232.0	33.0	68
Ayrshire butter:					
No. 17*	9	37.8	232.3	33.5	66
Breed unknown:					
No. 18	13.3	35.4	227.8	33.5	71
No. 19	12.9	41.2	222.3	33.0	208
No. 20	13.9	33.3	227.2	34.6	65
No. 21	14.1	33.8	227.8	33.5	65
No. 22	14.2	32.8	228.0	35.0	100
No. 23	13.4	33.3	228.5	34.4	88
No. 24	13.5	39.2	224.8	33.2	103
No. 25	14.1	31.9	228.0	35.0	62
No. 26	13.9	37.8	232.3	33.5	66
Average	13.9	35.6	228.2	33.8	93
Average for all	13.95	35.6	227.5	33.7	127

* Butter from the milk of a single cow.

An inspection of this table shows that the variations in all of the tests are greater than has been generally supposed. Especially is this true with those butters from the milk of a single cow. These variations are so marked that the application of any single test might fail to detect even 30 per cent. of adulteration. It is, however, unlikely that a pure butter will be found deficient in all respects, so that, in doubtful cases, the application of as many tests as possible is advisable.

The report of the committee was then taken up for discussion by sections.

PHYSICAL METHODS—MICROSCOPIC TESTS.

Dr. BABCOCK approved of the use of the microscope as a negative test—that is to say, with the selenite and polarized light a variegated

field would indicate something other than pure butter, although some butter which had been adulterated might show a homogeneous field.

Dr. JENKINS asked how "baled" butter would act, and was informed that it would give results like pure butter.

The section was then adopted.

SPECIFIC GRAVITY.

Dr. WILEY said 40° C. was selected instead of 37°. 7, as some substances, such as neutral lard, do not melt completely at the lower point.

Dr. JENKINS spoke of the use of the Westphal balance, and said: We have had considerable work to do in the examination of butter for our State dairy commissioner, and have found Westphal's balance extremely convenient for the purpose of taking specific gravity of the melted fat. The method is fully described and results given in our last annual report. The chief advantages of the method are rapidity of execution, accuracy, and the comparatively small volume of fat required.

Dr. WILEY replied that a large number may be done at one time in picnometers.

Several members then spoke of the possibility of calculating results obtained at 100° C. to 40° C. for comparison. A table of co-efficients of expansion was said to be given in Allen's Commercial Analysis.

The section was not modified.

MELTING-POINT.

The method of Wiley, as described by him, met universal approval and was not modified.

CHEMICAL METHODS—REICHERT'S PROCESS.

Dr. WILEY said that sulphuric could be used as well as phosphoric acid, if care was exercised, and was confirmed by Dr. Crampton. The following results were cited:

	P_2O_5.	H_2SO_4.
Volatile acids.....	15.39 15.90	15.75 And less.

Professor CALDWELL thought as many transfers as were described were dangerous and would prefer to saponify in flask with a long tube as condenser, completing all the operation in one vessel.

Dr. BABCOCK stated that he had found some difficulty in removing the alcohol, but by the use of strongest aqueous potash and adding only 3 or 4 cc. of alcohol, which is readily removed, he avoided trouble. The saponification is rapid.

Dr. JENKINS said he used the same method, since the alcoholic potash seemed to decompose in time and the results obtained were too high.

Dr. CALDWELL asks if a tube to act as condenser is necessary in this way of working.

Dr. JENKINS said he used one.

Dr. BABCOCK did not, since saponification was so rapid and complete before the boiling-point is reached.

It was then moved and adopted to substitute Dr. Babcock's method.

INSOLUBLE ACID.

The method described was adopted without modification.

SATURATION EQUIVALENT.

Dr. BABCOCK pointed out the necessity of having the alcoholic potash free from carbonate.

Dr. WILEY confirmed the statement, and said that saponification could only be completed with great difficulty in presence of carbonate.

The section was then adopted.

WATER.

Dr. WILEY said he found nothing necessary to assist in driving off water.

Dr. JENKINS found sand an assistance.

The section was adopted.

SALT, MINERAL MATTER, CURD.

These sections were adopted without change.

Dr. WILEY then suggested the addition of the method of *iodine absorption* as being simple and valuable.

A motion to do so was made and carried.

Dr. MCMURTRIE asked for the best method of separating the water and curd from the fat.

Dr. WILEY described his method as in use in the Department of Agriculture, as follows: The butter is melted and allowed to stand until the water and curd have settled. The supernatant liquid fat is then poured off through a dry filter placed in a hot water jacketed funnel.

Dr. JENKINS said he found, at times, when but little water is present, that the addition of a spoonful of salt was advantageous.

It was moved to introduce this in the official method, which was carried.

The report on butter was then recommitted, and the Convention adjourned to Wednesday morning, the 17th, at 10 o'clock.

WEDNESDAY MORNING.

The report on method of milk analysis being in order for discussion it was taken up by sections.

On the determination of water, Dr. JENKINS spoke as follows:

"We have found no method of determining water and fat in milk superior in accuracy or speed of execution to the sand method—that is, evaporating the milk with a known weight of dry sand to determine water, and extracting the pulverized residue with absolute ether in Johnson's, sometimes called Tollen's, extractor to determine fat. It has been abundantly proved that this method determines water with absolute accuracy. It has been recently objected by English analysts that the determination of fat by this method gives lower results than by Adams's method.

"Our own experiments have shown almost perfect agreement with Adams's method when the paper coils used in the Adams process were perfectly freed from wax or resin previous to use, an operation requiring time and care. If not previously extracted, of course the results by Adams's method will be higher."

Professor CALDWELL moved to substitute for the determination of water a method which he found desirable. One to two cubic centimeters of milk are to be evaporated on a water-bath in a watch glass, a few centimeters in diameter, for thirty minutes, and then dried at 100° C. in an oven for one hour.

Mr. FARRINGTON stated that he preferred 4 cc. and four hours, to which Professor Caldwell replied that the shorter exposure by his way of working was better, and that he used a water oven for drying.

Professor MYERS, in speaking of Professor Caldwell's process, said:

"I think that it is highly desirable that the method adopted should contemplate also the determination of one or more of the other ingredients of the milk, such as the total solids and fats and ash, or the total solids and the albuminoids from the same quantity of milk used for the determination of the moisture. Should we adopt the amendment proposed by Professor Caldwell we would be forced to weigh an additional quantity for the determination of the solids and fats or solids and albuminoids. We should constantly keep before us the question of economy of time, as that is often of importance to the State analysts."

Dr. WILEY called attention to indefinite boiling-points due to variations in altitude and indefinite temperature of water-drying ovens.

Professor SCOVELL suggested in consequence a definite temperature of 98° C.

After some further discussion the motion was carried.

The use of asbestus as a combined means of estimating moisture and fat as practiced in Canada was explained by Dr. MACFARLANE, who was

requested by the Convention to write a description for use as an alternate during the ensuing year.

The description is given among the official methods, Appendix A, p 76.

ALBUMINOIDS.

Dr. JENKINS referred to the determination of albuminoids by the Kjeldahl method, and said:

"Mr. Farrington has found trouble in digesting, by Kjeldahl's method, milk residues with the pulverized glass dishes referred to in the report, because of dangerous "bumping" in the liquid. We much prefer to weigh from 2 to 5 grams of milk directly into the digestion flask and proceed as with a solid substance. This method gives with us results which agree with those of the soda-lime method, and it has been thoroughly tested with satisfactory results in Germany, both with milk and with urine, where the per cent. of water runs up to 96."

A motion to introduce this method among those which are to be official was then carried.

FAT.

The question being raised as to the use of Adams's method for the determination of water as well as fat, the opinion seemed general that the results were not reliable.

Dr. WILEY described the Adams method as in use at the Department of Agriculture, and showed specimens of the paper used by Professor Adams, which he had recently received.

Several members stated that they had had difficulty in meeting with success, as the paper to be had in this country was either too fuzzy or contained considerable extractive matter not easily removed.

Dr. MCMURTRIE said that he had found in the paper used by him a waxy resinous substance, with difficulty soluble in ether but freely in 90 per cent. alcohol, but that Canadian asbestus had proved an excellent absorbent, and as it could be ignited did away with all danger of having foreign material soluble in ether present.

Professor SCOVELL called attention to his use of a tin-foil dish instead of the glass Schälchen for use in determining water and fat. The residue can be equally well extracted.

On motion this method was made an alternate with the Adams method.

Dr. WILEY moved that the Morse and Pigot method with anhydrous $CuSO_4$ be made an alternate, and this was agreed to.

Considerable discussion then ensued on the Macfarlane asbestus method, and Professor MACFARLANE explained that he dries the milk in current of hydrogen to within .1 per cent. and gets slightly higher results than with Adams' method.

On motion of Dr. WILEY the use of the lactocrite was also approved for those who have them.

It was then agreed that the alternates should be carefully compared and the results reported to the next meeting.

SUGAR.

Dr. FREAR desired some other method than the polariscopic, as some operators have no instrument, and after some discussion it was agreed that the method of extraction with alcohol and of determination with Fehling solution should both be inserted.

Professor SCOVELL found difficulty with the end reaction with Fehling.

Dr. MCMURTRIE said: The end reaction may be determined accurately by ferrocyanide and acetic acid. Saturate filter paper with ferrocyanide and acetic acid; spread upon a plate. When the standard solution has been added and while boiling take a drop upon a glass rod, and placing upon the saturated paper a clean piece of filter paper, press the rod with drop upon it. If a spot appears upon the saturated paper continue the titration until no spot appears.

ASH.

Dr. MCMURTRIE moved that a small quantity, 3 cc., of nitric acid be added to the milk before incineration, which was passed, and it was moved and seconded that the method proposed by Dr. Wiley be adopted as a whole.

Professor MYERS then called for the reading of the report, which was said at the time to be impossible. He remarked:

I called for a reading of the report as amended.

[*Scattering remarks from the members.*] Mr. MYERS. Mr. President, it is not in a factious spirit that I call for the reading of this report so that the members of the Association may vote upon it intelligently. I object to passing upon such important matters in undue haste. Ordinary business care should be taken.

Professors CALDWELL, CHAZAL, and others thought it not necessary to delay action, as the committee would perfect the report.

Professor MYERS. Mr. President, I do not desire to appear in this connection as expressing lack of confidence in the committee. On the contrary, I have the highest respect for, and confidence in, every member of the committee, but the point that I desire to insist upon is this, viz: We meet here as official chemists or as chemists whose work and whose methods must exercise a very important influence upon the commercial interests of this country. There is perhaps no other scientific organization in this country that has so much dependent upon the accuracy of its methods and the efficiency of their execution. The courts, the public, and the manufacturers are all more or less directly affected by our deliberations, and we owe it to ourselves as well as to the honor of the Association to which we belong that we should not act with unseemly haste. Now, we are asked to vote upon the adoption of a method of analysis about which even the secretary of the Asso-

ciation, the chairman of the Association, and the chairman of the committee disagree as to the changes made in the report by the Committee of the Whole. It is a bad precedent for us to adopt: it is calculated to bring discredit upon us personally and upon our organization. It does injustice to science and to the great interests involved. I therefore must respectfully insist upon due deliberation and proper form of report before the members of this Association be expected to vote upon the report of this or any other committee.

The motion was then withdrawn, the report recommitted, Professor Myers being placed on the committee to fill the vacancy caused by the absence of Professor Armsby.

Mr. CHAZAL then asked unanimous consent of the convention to listen to a communication from Mr. A. De Ghequier, representing the National Fertilizer Association, which was given.

Mr. DE GHEQUIER spoke as follows:

Mr. PRESIDENT AND GENTLEMEN OF THE OFFICIAL AGRICULTURAL CHEMISTS' ASSOCIATION: On behalf of the Committee on Analysis of our Association I have the honor to submit to you for favorable consideration, and such action as you may feel justified under the circumstances to take, several requests, all of which, it is believed, fall entirely within the scope of your Association. The circular issued by this Association on July 28 acquaints you with the results of an investigation made for the purpose of testing the accuracy of the present method of analysis. It will suffice for me to state that the same goods, represented as sample 9982, received at first less than 8 per cent. soluble, and less than 13 per cent. available, phosphoric acid, and this analysis was published as correctly representing the goods in question. I do not know that I can improve, as regards what the Association is anxious to obtain from you, upon the expressions made by the chairman of our Committee on Analysis. In his correspondence with me on the subject in a letter written May 7 he says: "I think we should strive to get the chemists to agree to certain conditions of sampling and analyzing, in order to give the manufacturers some protection, whereas at present they have little or none. The following points, it seems to me, are important in this direction: The sampling should be done from a representative number of packages in each lot, and not, as is the case in many instances, from a single bag of 100 or 200 pounds. The sample should be drawn from different portions of the sack, that is to say, not be a sample from the top; and, then, in drawing the final sample from the mixture of all the samples, great care should be used to get a representative one. Duplicates should in all cases be held by the chemists subject to the order of the manufacturer."

Another important point in sampling is this: Samples should always be drawn without any knowledge on the part of the manufacturers that such sampling is done; that is, at points where there will be no chance of the manufacturers having any knowledge as to the sampling being

done. In other words, every precaution should be taken to get representative samples of each manufacturer's goods as found on sale in the open market. The analysis question is still more important. Uniform methods for determining all elements should be agreed upon and strictly adhered to, and all analyses should be made in duplicate, and, where possible, by different methods, as a check. The analyses should in all instances be reported to the manufacturers whose goods are sampled before they are published, in order to give the manufacturer a chance for protest. The protest need not necessarily affect the station's decision about the publication of the analysis, unless sufficient grounds are furnished for the objection, but a chance for protest should certainly be given to the manufacturer. Where such a protest is made the director ought certainly be willing to go over the results in person, as oftentimes, I believe, errors are made by careless or incompetent assistants. Where nitrogen is claimed to be present in the form of nitrate of soda each chemist should certainly not only be willing but ought to be bound to analyze by the absolute method, or by such a method, at any rate, as will determine nitrogen in the form of nitrates. We are constantly having trouble from this source, the chemists not giving us sufficient nitrogen. I do not suppose that the time has come when chemists will consent to give up valuations, but it should come soon, as there is certainly no justice whatsoever in placing valuations upon each manufacturer's goods when the chemists cannot, by any possibility, tell what the real agricultural or intrinsic value of the goods in question is. Determining, as they do, simply the chemical elements of plant-food, the chemists cannot on this alone compute a value on any fertilizer that is fair to the manufacturer, for some materials are valuable as plant-foods, and consequently high priced, while others are of very little value, and consequently of very low price. Then there is nothing ascertained as to the mechanical condition of the fertilizer, its dryness, fineness, the thoroughness with which it is mixed, &c., all points of the greatest importance to the farmer. Also there is no credit given for the proportions of the elements found in the fertilizer. A fertilizer containing a very large proportion of nitrogen and very little potash or ammonia would be given the same valuation by a chemist, if it were used for a potato or corn crop, as one containing the proper proportions of the three elements required for these crops. All these points are, I think, of great importance, and should be properly weighed by the directors of the State Experiment Stations in making up their reports of the various fertilizers inspected by them. As it would be impossible for them to correctly take into consideration in their valuations these elements, they should not give valuations, but merely the guaranteed composition of the fertilizers and their own analyses, for comparison. Another point against valuations is that they tend to keep the farmers and the public in ignorance as to the relative merits of chemical fertilizers, based upon their composition, whereas the dis-

continuance of the publication of valuations would oblige the public to scrutinize the analyses closely, the percentages of the different elements found in each fertilizer, compared with the guaranteed percentages, and the relative merits of one fertilizer as compared with another, based on the relative proportions of the different elements existing in each. In other words, to discontinue publishing the valuations would tend to educate the farmers, as they would no longer, as they do at present, merely compare with each other the valuations given the various fertilizers. I think it would be well for the Association to present to the chemists a statement embodying some of these views with others that are important, and then make certain requests as deductions from these arguments, to be adopted and strictly adhered to by all the Association chemists in their inspection of commercial fertilizers.

Following is the circular of the Exchange:

THE NATIONAL FERTILIZER ASSOCIATION,
Baltimore, July 28, 1887.

Complaint having been made to the committee on analysis about very unsatisfactory, because exceedingly low results of the official analyses in the State of Ohio, the committee on analysis decided to institute a test case and selected for that purpose two kinds of acid-phosphate, one being dissolved land, the other dissolved river-rock. The co operation of the State Board of Agriculture had been secured beforehand, and the secretary of said board drew in person samples from each lot which were by him divided, sealed, and forwarded by express to the following chemists: Professor Lord, of Ohio; Professor Kedzie, of Lansing, Mich.; Professor Johnson, of New Haven, Conn.; Professor Wiley, chemist to the United States Department of Agriculture and to the National Fertilizer Association This last sample was once more subdivided and expressed to Dr. Chas. W. Dabney, of Raleigh, N. C.; and sent to Prof. Wm. Simon, and the manufacturer of the goods.

The results are herewith submitted as follows:

	Sample No. 9982.					Sample No. 10720.				
	Soluble.	Reverted.	Insoluble.	Available.	Total.	Soluble.	Reverted.	Insoluble.	Available.	Total.
1	10.35	3.18	2.29	13.53	15.82	10.38	2.79	1.19	13.17	14.36
2	9.76	3.29	2.67	13.04	15.71	9.92	2.78	1.72	12.70	14.42
3	12.00	1.59	2.34	13.59	15.93	9.50	2.53	1.90	12.03	13.93
4	9.97	3.62	2.10	13.49	15.59	10.09	2.90	1.40	11.99	14.39
5	10.50	3.05	2.54	13.55	16.09	10.71	2.76	1.40	13.47	14.86
6	11.06	3.09	2.12	14.15	16.27	11.57	1.63	1.53	13.20	14.73
7	10.85	2.30	3.22	13.15	16.37	10.83	2.44	1.98	13.27	15.25

It will be seen that the two analyses marked No. 1 and No. 5 agree very closely and represent the fair average between highest and lowest results. But if the analysis No. 3, in both cases, is analyzed, it is apparent that the same chemist who found the least percentage of soluble in one sample found the highest in the other, and the 12.03 available of sample 10720 form a strong contrast to the 13.47 obtained by another chemist of the same sample. The difference calculated as commercial value shows the same article to be of too different value to be classified as identically the same article, yet on analyses differing thus widely a manufacturer's reputation rests without any redress whatsoever. Who is responsible for the injury done a manufacturer's

goods by the publication of analyses which often are proven erroneous "*post festum?*" It would seem a matter of justice to the manufacturer to accord him, in all cases where the official analyses fall below the standard claimed, an opportunity to verify its correctness. After publication of an erroneous analysis, the mischief wrought cannot be undone. The members are therefore requested to express to the secretary their views on this important matter prior to August 10, proximo, so that the committee may be enabled to lay before the next meeting of the Official Chemists' Association, which will be held in Washington, on August 16, a memorial embodying the views of the members.

It is desirable, also, that members, whose business suffered actual injury from the premature publication of incorrect official analyses, should put the association in possession of such facts, so that the necessity of a much needed reform of the present prevailing practice may be illustrated in the most forcible manner possible.

The committee are of the opinion that imperfect sampling has much to do with the unsatisfactory results which are still of too frequent occurrence. It is also believed that the inability of the chemists to make all analyses in person accounts for many serious discrepancies. At the same time the committee is of the opinion that where a manufacturer's reputation is at stake, and grave injury done to his business, an inexperienced assistant is not the proper person to attach an arbitrary value to goods on the strength of what is often an inaccurate analysis.

For the Committee on Analysis,

ROBERT S. BRADLEY, *Chairman.*
A. DE GHEQUIER, *Secretary.*

Professor LORD then made some remarks in regard to the case, and Mr. CHAZAL moved that the communication be referred to a committee of three, with the chair as *ex officio* chairman.

This motion was seconded and carried, and the chair appointed Messrs. Chazal, Gascoyne, and Lord upon the committee.

The Commissioner of Agriculture then expressed his regrets to the convention at being unable to address them at length, and his pleasure at seeing the Association in such a prosperous condition. He desired to do all that he could to assist them.

Dr. MCMURTRIE then asked unanimous consent which was granted, to present a communication from the State Board of Agriculture of Illinois.

Dr. MCMURTRIE stated that in his letter of instruction from the State Board of Agriculture of Illinois he was directed to urge the appointment of a committee to prepare uniform blanks for use of all interested in the inspection of commercial fertilizers, so as to insure, as far as possible, uniformity of procedure. The same board also request that the Association determine upon a definition of the term "commercial fertilizer." He also presented copies of the Illinois law and of blanks adopted according to the conditions thereof.

Professor SCOVELL moved to refer the request to a committee of three, which was seconded and carried.

The chair appointed Messrs. McMurtrie, Stubbs, and Scovell.

The Dairy Committee then read the official methods, as agreed upon, for the ensuing year, and they were accepted as a whole*

The convention then adjourned till 1.30 p. m.

* See appendix A.

WEDNESDAY AFTERNOON.

The Convention was called to order at 1.30 p. m., and in the regular order listened to the report of the Committee on Phosphoric Acid, presented by Professor Stubbs, chairman.

REPORT OF THE COMMITTEE ON PHOSPHORIC ACID.

The Committee on Phosphoric Acid respectfully submits the following:

It has been customary for previous committees to divide their reports into three parts: (1) Brief notices of new analytical methods for determining P_2O_5 proposed during the year; (2) Results of co-operative work done by the committee and members of the Association; (3) Recommendations for the next year.

I. The very full reports previously made and the almost complete absence of new literature on P_2O_5 during the past year renders our duty under the first head short and decisive. We know of no new methods worthy of recording.

II. Early in the fall of 1886 your chairman obtained samples of English and South Carolina acid phosphates. The first was selected from a cargo of 1,100 tons, imported in bulk by the Planters' Fertilizing Company of New Orleans, and was guaranteed to contain 12 per cent. soluble P_2O_5. The other was donated by Etiwan Phosphate Company of Charleston, S. C.

A few weeks later the Navassa Phosphate Company of Wilmington, N. C., placed at our disposal a sample of their acid phosphate. These samples, so generously furnished by the manufacturers, were ground in a mill until they all passed through a sieve eighty meshes to the inch; then thoroughly mixed and smaller samples sent to twenty-one analysts with request to analyze by the methods prescribed by this Association at its last meeting. Other methods of analysis were requested. These samples were lettered *a*, *b*, and *c*.

a.—South Carolina acid phosphate.

b.—English acid phosphate.

c.—Navassa acid phosphate.

Samples were subsequently sent to several commercial chemists of Charleston and Baltimore upon application.

Unfortunately returns have been received from only six laboratories, results of which are herewith appended.

Results of phosphoric acid determinations.

Analyst.	Moisture.	Soluble.	Reduced.	Insoluble.	Total.	Available.
Sample a—						
A	18.14	5.58	4.45	2.60	12.63	10.03
B	18.22	5.83	4.37	2.40	12.60	10.20
C		6.04	4.09	2.41	12.54	10 13
D	20.96	6.00	4.11	2.38	12.49	10.11
E	17.25	6.10	4.65	2.35	13.10	10.75
F	18.10	6.15	5.36	2.17	13.68	11.51
Sample b—						
A	15.03	9.38	2.17	.43	11.98	11.55
B	15.07	9.48	2.24	.43	12.15	11.72
C		9.31	2.23	.20	11.74	11.54
D	16.15	9.34	2.41	.35	12.10	11.75
E	15.65	9.35	2.90	.85	13.10	12.25
F	12.85	9.26	2.81	.50	12.57	12.07
Sample c—						
A	6.42	9.63	5.17	4.73	19.53	14.80
B	6.31	9.53	6.06	4.11	19.70	15.59
C		10.81	4.78	3.71	19.30	15.59
D	7.31	8.57	6.88	4.06	19.51	15.45
E	5.40	8.60	6.49	4.25	19.25	15.00
F	7.45	8.78	6.68	4.32	19.78	15.46

Averages.

	No. of determinations.	Mean.	Highest.	Lowest.
Sample a—				
Moisture	5	18.53	20.96	17.25
Soluble	6	5.95	6.15	5.58
Insoluble	6	2.38	2.60	2.17
Citrate, soluble	6	4.50	5.36	4.09
Total	6	12.84	13.68	12.49
Available	6	10.45	11.51	10.03
Sample b—				
Moisture	5	14.95	16.15	12.85
Soluble	6	9.35	9.48	9.26
Insoluble	6	.46	.85	.20
Reduced	6	2.46	2.90	2.17
Total	6	12.27	13.10	11.98
Available	6	11.81	12.25	11.54
Sample c—				
Moisture	5	6.58	7.45	5.40
Soluble	6	9.32	10.81	8.57
Insoluble	6	4.19	4.73	3.71
Reduced	6	5.99	6.68	4.78
Total	6	19.51	19.78	19.25
Available	6	15.31	15.59	14.80

The following may be noted in regard to the above:

(1) The determinations of moisture are, in our opinion, too varying. In *a*, *b*, and *c*, respectively, the differences between the highest and lowest results are 3.71, 3.30, and 2.05 per cent. Upon a 12 per cent. acid phosphate a difference of 3 per cent. in moisture may mean as much as .4 per cent. of phosphoric acid. An examination of results will show that the variations in moisture are not always attended by variations in P_2O_5, a fact which shows that the variations are due rather to personal errors than to differences in samples.

(2) The determinations of total phosphoric acid are in the main satisfactory. In *a* five out of the six are within .33 per cent. of the average, the sixth one .84 per cent. of the average; four out of the six are practically the same, the average being 12.56 per cent.; the lowest, 12.49 per cent.; the highest, 12.63 per cent.

In *b* the same remarks are applicable. Two analysts, getting higher results, elevate the average, while, without their work, the average is 11.99 per cent., the highest 12.15 per cent., and the lowest 11.74 per cent.

In *c* the results are more uniform.

(3) The determinations of soluble P_2O_5 alone are very satisfactory in all but *c*. In this sample the average was 9.32 per cent. Highest, 10.81 per cent.; lowest, 8.57 per cent.; difference between highest and lowest, 2.24 per cent. Since no such differences exist in the available, the impression is created that with the lower results the exact directions of the method of determining soluble P_2O_5, so far as extracting it with water on the filter is concerned, have not been followed. In all acid phosphates containing iron and alumina it is absolutely necessary to wash on the filter with small quantities of water at a time and letting each addition run off before another is made. A small quantity dissolves while a large one precipitates, which being caught on the filter is carried into the reduced. In States where the soluble P_2O_5 is given a higher value than the reduced, care here is very necessary to secure justice to the manufacturers. In States where both soluble and reduced are classified as available and together assigned the same value no such injustice will be done.

(4) The insoluble determinations are on the whole satisfactory. In *a* the average being 2.38 per cent.; highest, 2.60 per cent.; lowest, 2.17 per cent.

In *b*, when the insoluble is very small, larger proportional discrepancies exist. Average, .46 per cent; highest, .85 per cent.; lowest, .20 per cent. In *c* the average is 4.19 per cent; highest, 4.73 per cent.; lowest, 3.71 per cent. A difference between the highest and lowest, 1.02 per cent.; an amount too great in scientific work.

The above discrepancies may result from a different interpretation of the prescribed method, which says: Wash the residue of the treatment with water into 150 cc., flask with 100 cc. of strictly neutral ammonium citrate solution of 1.09 density, shed and add the filter paper, cork the flask securely, *place in a water-bath with constant temperature of* 65°*C. and digest for* thirty minutes at this temperature with frequent shakings, &c. The following letter from Dr. Dabney, of North Carolina, first calling our attention to this point is here given in order to show how two different interpretations may be placed upon the wording of this method and different results obtained thereby.

RALEIGH, *March* 26, 1887.

MY DEAR SIR: As chairman of the Phosphoric Acid Committee of the A. O. A. C., I beg to call your attention to the great importance of a better defined method for the digestion with ammonium citrate.

The way it reads now a man may try fairly to follow the method and still get results considerably at variance with those of another trying equally as hard to do right.

The words (see p. 49 of last published methods, paragraph 4) "place in a water bath with constant temperature of 65° C. and digest for thirty minutes at this temperature with frequent shakings" are susceptible of too varied interpretation. We have heretofore interpreted this as meaning "Warm the bath to 65° C., place the flask in this bath for thirty minutes," &c.; others, as Dr. Jenkins, if I understand him, hold that it means, "Place the flask in a cold bath and heat to 65° C., then count thirty minutes from time it reaches 65° C., &c. Others think time ought to be allowed for contents of flask to reach 65° C., &c.

These different ways of treating different phosphates make differences varying from one-half to one and one-half per cent. on the available.

Take the case of our and Jenkins's ways and compare them.

A given fertilizer gives us 4.18 per cent. insoluble, working our ways; Jenkins's way, gives us 3.59 per cent. of insoluble; or, quoting from Mr. Battles's report to me—

"This insoluble was placed in cold bath and heated with one lamp, with following results:

Bath.	Time.
18° C	10.15
30	10.20
45	10.30
30	10.40
65	10.45

"Digestion was continued for thirty minutes, or till 11.15. Flask was shaken every five minutes from 10.15.

"We see then that the flask remained thirty minutes more than usual, and this at a temperature averaging that in the old Washington method.

"The method à la Jenkins gave .59 per cent. P_2O_5 less than that before.

"To have a quantitative method with no more definite directions than the above is to my mind utterly foolish, and well calculated to bring discrepancies into results."

Yours truly,

CHAS. W. DABNEY, JR.

Prof. W. C. STUBBS,
Kenner, La.

We quite agree with Dr. Dabney, and here recommend that this clause be so amended as to convey the exact meaning of this Association.

III. Your committee recommends, in conclusion, the continuance of the present general method for the determination of phosphoric acid, subject, of course, to such minor modifications as your wisdom may determine. It would, besides the needed amendment already alluded to, call particular attention of the official chemists and

their assistants to the necessity of following in the minutest detail whatever method may be adopted by this Convention, in order to gain the most perfect unanimity of results, thus securing the confidence of the public in our work.

IV. Since most of the members of this Association are, or will be, connected with experiment stations under the operation of the new Hatch bill, which we hope will be available by the beginning of another year; it may not be inappropriate to again call attention to the experiments of Dr. Paul Wagner to establish by soil tests the relative manurial value of the different forms of phosphoric acid, and to devise analytical methods for determining in the laboratory what will be the approximate manurial values in the field. Accurate experiments of a most extensive nature are required before we can render agriculture the assistance which is expected of us. To answer a farmer that we can tell the commercial but not the agricultural value of a fertilizer is a confession that brings grief, since the agricultural value, after all, gives the commercial. This question is made more serious recently by the introduction into our markets of the phosphate slag, a by-product in the Thomas-Gilchrist process of making steel. This basic slag is on our market, and both the manufacturer and farmer are demanding of us to assign it its proper place in our agricultural wealth. A sample taken from a lot recently imported into New Orleans from Glasgow, Scotland, gave, on analysis by official method: Reduced phosphoric acid, 5.37 per cent.; insoluble phosphoric acid, 11.14 per cent.; total phosphoric acid, 16.51 per cent. This slag is said to have a high agricultural value, while our methods of analyses show only one-third available.

These questions must be solved, and it seems to your committee that the present is a most opportune time for the adoption of plans looking to concerted action in the conduct of such experiments as will ultimately throw light on this important subject.

WM. C. STUBBS, *Chairman.*
W. E. MOSES.

The report being open for discussion, Professor LORD called attention to the indefinite directions as to the manner of heating the citrate solution during its action on the phosphate. He had received quite different directions from two members in reply to an inquiry for interpretation of the method. One had stated that the flask containing the citrate was to be placed in water which was to be rapidly raised to 65° C., and the other that the water was to be at 65° C. when the flask was put in.

After some discussion the directions were modified so as to read as they appear in the appendix.

Professor FREAR objected to the term "gentle heat" instead of "boil gently."

Professor LORD suggested that the form of flask and its size were important, and proposed an Erlenmeyer flask holding 200–240 cc.

The question of the use of a filter-pump in filtering the precipitate was brought up, and it was agreed to be desirable and necessary.

Professor GASCOYNE inquired as to whether more definite directions should not be given as to the preparation of the sample. He uses a 20-mesh sieve and a tight tin box; Dr. Jenkins uses a 25-mesh. The necessity for tightness of sifting-box was due to the ease with which moisture is gained or lost, depending, as Professor Stubbs showed, on the climatic conditions under which different chemists worked. In Louisiana the least exposure to the air is accompanied by gain.

The use of hydrochloric acid and chlorate met with opposition from many members, and called out some positive opinions.

Dr. JENKINS found it especially bad with castor pomace, and several chemists were unable to get all the P_2O_5 in solution in this way.

Professor MYERS said:

The working of the present method of determining the total P_2O_5 by HCl and $KClO_3$ in my hands is highly unsatisfactory. It has been next to impossible for me to extract all of the P_2O_5 by this method. I have found in most cases that more P_2O_5 can be obtained by the use of $Mg(NO_3)_2$ than by the method at present in force.

Personally I am in favor of returning to the use of the $Mg(NO_3)_2$ as the method for future use for the oxidation of organic matter.

I have never had the trouble with this reagent complained of by other gentlemen. The evaporation and burning off both proceed quietly, without bumping or spirting, and the process is free from the difficulties which follow the use of HCl and $KClO_3$.

I grant freely that in the hands of other chemists the present system may be a success, but am quite positive that at least, so far as I am concerned, a change is desirable. It strikes me that it is desirable to allow the chemist considerable latitude, as there is a variety of fertilizing materials in our markets, and it may not be possible in many cases to secure correct results. Besides this, as we are fully aware, a process successfully carried out by one person or in one laboratory may present serious or almost insurmountable difficulties to another. From some cause—you may call it the personal coefficient if you wish—it is next to impossible for chemists to secure concordant results by different methods. One can excel by the use of one process while another may be forced to employ a different method to secure equally correct results. Whether this be due to the personal error of the chemists, or to the impossibility of having equally accurate apparatus in all laboratories, or to both, is not important at present. The fact remains and we should not ignore its existence.

I am, therefore, most heartily in favor of giving more latitude in the methods used for destroying the organic matter and for estimation of the total P_2O_5.

This can be accomplished by permitting optional methods, which is certainly desirable.

Professor SCOVELL moved that the use of nitric acid be substituted for HCl and $KClO_3$.

Professor STUBBS moved to amend by the use of HCl and $MgNO_3$.

Liberty of action in the matter seemed to be most desirable, and the motion, amended to the use of either solvent, was passed, and in consequence the section containing this provision was ordered to be somewhat modified.

Mr. CHAZAL desired the shredding of the first filter paper to be dispensed with, and it was agreed to as unnecessary, but the rubbing up

of the acid phosphate with small quantities of water for the extraction of soluble, especially on high-grade goods, was deemed to be advisable.

The report was then recommitted.

Dr. McMURTRIE then presented, by unanimous consent, the report of the committee to consider the communication of the Illinois State Board of Agriculture, as follows:

Your committee, to whom was referred the matter of the request of the State Board of Agriculture of Illinois, that this Association adopt (1) "a uniform series of blanks for use of all interested in inspection of commercial fertilizers," and (2) "a clearly expressed definition of the term commercial fertilizer," have to report that it appears that in the conditions of the laws of the different States it is impracticable for this Association to take action upon these points. Only methods for taking samples, making analyses, and stating results can be recommended with any satisfaction to all concerned.

WM. McMURTRIE.
WM. C. STUBBS.
M. A. SCOVELL.
Committee.

Professor CHAZAL desires that something should be done for uniformity in branding sacks, form of labels, blanks, method of drawing samples.

Dr. McMURTRIE said one year's experience in Illinois had shown a necessity for it.

Professor MYERS thought the matter not in the scope of the Association, and that no attempts should be made to influence legislation.

Dr. CHAZAL moved that the report be recommitted, with instructions to the committee to take into careful consideration the matters proposed and report at the next annual meeting.

He stated that he believed that the subjects proposed merited a more careful consideration by the Association than it had been possible to give them.

That the objection made, that such an inquiry was beyond the scope of the Association, which had no power to compel State legislatures or boards of control to adopt its recommendations, would apply with equal force to the consideration of methods of analysis. That the Association did not have any power to enforce the adoption by any State control of its methods of analysis; it only gives to certain methods the stamp of its approval and recommends their adoption for use.

That correct methods of sampling are certainly as essential as correct and uniform methods of analysis to the attainment of uniform results.

If any of the subjects proposed for the consideration of the Association should, after careful deliberation, be considered by the committee to be beyond the scope of the Association, it might so report, with a statement of its reasons for such action.

That the object and effect of the motion was not to commit the Association in any way, but merely to have these matters settled definitely, once for all, after careful consideration, and thus avoid the necessity of dealing with the subjects in part year after year.

Professor MACFARLANE said that Canada was laboring under the same difficulties, and that he would like to see some action.

It was moved and seconded to recommit the report for consideration by the committee, who are to report next year. Passed.

Mr. RICHARDSON then moved, and it was seconded and adopted, that all committees on method of analysis should recommend forms of statement.

The regular order of business having been taken up,

Mr. RICHARDSON, chairman, read the report of the Committee on Potash, as follows:

REPORT OF THE COMMITTEE ON POTASH.

In the absence of the remaining members of the committee on potash I have the honor to present the following report of the work which has been accomplished during the past year:

In the winter five samples of potash salts and potash fertilizers were distributed to members of the Association and others interested who had previously informed the committee that they would undertake the work. They consisted of—

(1) Chemically pure potassic chloride.
(2) Pure commercial potassic sulphate.
(3) Commercial muriate.
(4) Kainite.
(5) Superphosphate with potash.

In reply results were received from nine laboratories and eleven analysts, as follows:

Analyst.	No. 1.		No. 2.		No. 3.		No. 4.		No. 5.	
	Official.	Gladding.	Official.	Gladding.	Official.	Gladding.	Official.	Gladding.	Official.	Gladding.
A^1	.....	.....	.....	.....	.....	.....	13.70	.....	2.93	.....
A^2	.....	.....	.....	.....	56.76	.	13.73	.....	3.12	.....
A^3	.....	.....	.....	.....	56.24	.....	13.44	.....	2.96	.....
B	.....	.....	.....	.....	56.68	56.10	13.62	.....	2.87	2.90
	.....	.....	.....	.....	56.68	56.15	13.95	13.24	2.98	3.04
	.....	.....	.....	.....	57.08	.....	12.95*	.....	.....	.....
	.....	.....	.....	.....	.....	.....	13.07*	.....	.....	.....
C	.....	.....	.....	.....	.....	56.57	.....	14.06	...	3.09
	.....	.....	.....	.....	.....	56.66	.....	14.00	.	3.19
D	.....	...	...	.....	55.94	55.72	13.56	12.92	2.98	2.71
	.....	...	...	.....	55.88	55.73	13.16	13.16	2.97	2.74
E	63.10	63.14	53.65	53.68	53.89	54.25	13.60	13.35	2.65	2.75
F	63.14	63.17	54.02	54.04	56.44	56.48	13.35	13.45	2.71	2.70
G	62.89	62.99	53.92	53.99	58.22	56.96	13.68	13.87	3.18	3.27
H	62.61	62.45	53.66	53.60	56.65	56.87	.....	.....	.....	.....
I	.....	.....	54.04	.....	.....	.....	.....	.....	2.76	.....
Highest	63.14	63.17	54.04	54.04	58.22	56.96	13.95	14.06	3.18	3.27
Lowest	62.61	62.45	53.65	53.68	53.89	54.25	13.16	12.92	2.65	2.70
Mean	62.94	62.94	53.86	53.83	56.37	56.15	13.58	13.49	2.93	2.93
Difference	.53	.72	.39	.36	4.33	2.71	.79	1.14	.53	.57

*Washed with Gladding's solution.

These results, while showing close agreement among a few of the analysts, contain figures in other cases which are far from what they should be. It would perhaps be fair to state that the latter were the work of persons not as well up in potash determinations as some of our older members and to express regret that many of our best analysts did not contribute any work. The averages, I believe, may fairly be considered as correct or close approximations to the true amount of potash in the specimens, and it will be seen that a majority of the analysts are in agreement with these figures and that the extremes which are so far apart occur in only two or three in-

stances; still at the best the results hardly present the accuracy desired. The necessity for careful investigation on the part of each analyst into his method of manipulation and repetition of the analyses in cases where the results do not agree with the averages are suggested as the best means of improving our work.

The Lindo-Gladding method has proved extremely satisfactory and seems to be fast taking the place of the more tedious method previously in use. Its recognition by the Association as the standard for the ensuing year is recommended with retention of the old method for cases where the former seems inapplicable.

No information of importance has appeared in the journals during the past year as far as the committee are aware, and no suggestions have been communicated to them by members of the Association. They can only recommend, as has been said, the retention of the principles applied during the past season.

CLIFFORD RICHARDSON,
Chairman.

Dr. WILEY then read the following abstract of a method for the determination of potash lately adopted by the French Association of Chemists:

ABSTRACT OF THE REPORT ON THE ESTIMATION OF POTASH BY THE COMMITTEE OF THE FRENCH ASSOCIATION OF SUGAR CHEMISTS.

The sample of fertilizer being properly prepared, 10 or 50 grams are taken, according to the percentage of potash which it is supposed to contain. The sample is subjected to ignition at a low temperature for the purpose of driving off the ammonia and to destroy organic matters. If the fertilizer is a liquid and contains ammonia, instead of submitting it to calcination there is added to it 2 to 3 grams of caustic magnesia, and it is then boiled until reduced to half its volume.

The calcined sample is dissolved in hot distilled water, decanted upon a filter, and washed until exhausted. After the washings are cooled the volume is made up to 500 cc., or to a liter. Take of this solution a quantity representing 1 to 2 decigrams of potash, make the solution slightly acid with hydrochloric acid to transform the bases into chlorides. Add then, drop by drop, a quantity of bichloride of platinum, sufficient not only to precipitate the potash but also the soda, the lime, the magnesia, the baryta, and the strontia which may be present. The analyst need not bother himself about other bodies present, such as sulphuric acid, phosphoric acid, silica, alumina, iron, &c. The liquid is evaporated on a sand bath, in a porcelain dish, to a pasty consistence. After cooling, the mass is digested for a few hours with a mixture of 95 per cent. alcohol and ether, 9 parts of alcohol to 1 of ether. The mass is carefully rubbed with a pestle in order to insure the complete solution of the chloro-platinates of soda, lime, magnesia, &c. Afterwards it is washed upon a smooth filter with the same mixture until the washings are perfectly clear and colorless. When the precipitate of the chloro-platinates, collected upon the filter, is partially dried, the vessel containing the wash water is removed, and there is placed under the funnel a porcelain dish, under which a lamp is placed. The precipitate is now treated with boiling distilled water until the chloro-platinate is completely dissolved. What remains upon the filter paper is ordinarily silica or iron.

In a porcelain dish there is placed a solution of formiate of sodium in sufficient quantity to reduce all of the platinum of the bichloride employed. This solution is carried to the boiling point, and is poured slowly into the boiling solution of the chloro-platinate upon which the formiate at once reacts, reducing the platinum to the metallic state in the form of a black powder. Afterwards a few drops of chlorohydric acid are added to the liquid, and it is boiled for a few moments in order to concentrate it and to agglutinate the platinum. The liquid is decanted upon a smooth filter, which gives but little ash on incineration. The metallic platinum is washed

upon the filter paper with boiling water until it contains no chloride, which is ascertained by treating a drop of the filtrate with silver nitrate.

The chloro-platinate may also be reduced by hydrogen, and the platinum obtained by this method is easier to wash and dry than by the method just given. The impure chloro-platinate of potassium is introduced into a small glass tube drawn out at one end. This tube is fitted to a hydrogen apparatus, the current of hydrogen being carefully dried. When the air is all driven out, the tube is lightly heated. The chloro-platinate is reduced and the tube contains a mixture of platinum sponge, chloride of potassium, and some other foreign matters precipitated by the alcohol at the same time with the chloro-platinate. The mixture is washed several times, dried, and weighed. The increase in the weight of the tube gives the weight of the spongy platinum derived from the decomposition of the chloro-platinate, and from this is easily determined the weight of potash contained in the sample analyzed.

REAGENTS EMPLOYED IN THE ESTIMATION OF POTASH.

Bichloride of platinum.—This solution should contain 10 grams of the platinum salt in each 100 cc.

Solution of sodic formiate.—This solution should contain 10 per cent. of the formiate; 35 cc. should be taken for each 10 cc. of the bichloride of platinum employed. The weight of platinum multipled by .4786 gives the weight of potash.

Remarking on the report of the committee, almost all the members expressed their satisfaction with the Lindo-Gladding method.

Mr. FARRINGTON had found it would not work with wood ashes.

The report was then adopted making the Lindo-Gladding the leading method for the ensuing year, and the convention adjourned until Thursday at 10 a. m.

THURSDAY MORNING.

In the absence of the president the convention was called to order by the secretary, who nominated Professor Lupton as temporary chairman, which motion was seconded and carried.

The report of the Committee on Nitrogen being in regular order, it was delayed by unanimous consent to listen to the report of the committee to whom the communication of Mr. de Ghequier was referred.

The committee reported as follows:

REPORT OF THE COMMITTEE ON THE COMMUNICATION OF MR. DE GHEQUIER, SECRETARY OF THE NATIONAL FERTILIZER EXCHANGE.

Your committee have considered the communication of Mr. de Ghequier, secretary of the National Fertilizer Exchange, and beg leave to present the following report:

(1) That as regards uniform and accurate methods of sampling, your committee recognizes the great importance of the question, and recommends that it be referred to the committee already appointed for the consideration of the same subject.

(2) That as regards the use of uniform methods in determining nitrogen where nitrates are present, such methods have already been adopted by the Association.

(3) That as regards the other points suggested, report of analysis to manufacturer prior to publication, the revision, where desired, in cases of protest where such revision has not been previously been made, your committee would suggest that such a course of action, which is already practiced at many stations, is eminently desirable, and

should be commended to the attention of any of our members who have not been in the habit of following it.

(4) That it seems to your committee that it is somewhat doubtful whether the question of valuations is a subject within the scope of the Association, and your committee would therefore suggest that the matter be referred to the committee previously mentioned.

(5) In regard to the circular which has been offered as a part of the communication, your committee cannot see how the Association is concerned in questions of this character. But that as regards the particular matter it referred to, your committee would suggest that from the facts which have been brought out it is evident that the determinations in question were not made on the same sample, and it is further doubtful whether the two samples were representatives of the same lot of goods.

E. H. JENKINS.
P. E. CHAZAL.
W. J. GASCOYNE.
Committee.

The report was received and adopted.

Mr. CHAZAL moved that a committee be appointed by the chair to nominate officers for the ensuing year. This motion was seconded and passed, and the chair named Drs. Wiley, Scovell, and Gascoyne.

The Committee on Nitrogen then reported as follows:

REPORT OF THE COMMITTEE ON NITROGEN.

As required by the constitution of the Association, the Committee on Nitrogen herewith presents its report. The report covers (1) a brief notice of new methods for the determination of nitrogen proposed during the year; (2) results of work done during the past year by the committee and members of the Association, and (3) recommendations for the next year.

NEW METHODS FOR THE DETERMINATION OF NITROGEN.

Jodlbaur (Chem. Centralb., **17**, 433, J. S. C. I., **5**, 510) finds that when benzoic acid is employed in place of sugar as in the Kjeldahl method, when nitrates are present, as recommended by Asboth, the results are far too low. The author bases his method on an observation by Kjeldahl, that in his process the nitrogen in amines is readily converted into ammonia. The nitric acid is first converted into a nitrophenol, and subsequently reduced by zinc dust. For this purpose 0.2 to 0.5 gram of the substance is treated with 20 cc. of concentrated sulphuric acid and 2.5 cc. of phenolsulphonic acid (100 cc. contains 50 grams of phenol) and then with 2 to 3 grams of zinc dust and a few drops of platinic chloride solution. The action is complete in about four hours. It is more rapid when phosphoric acid is mixed with the sulphuric acid.

Stutzer and Reitmaier (Rep. Anal. Chem. **7**, 4-6) finds that the method of Jodlbaur does not give good results when the manure contains a large percentage of nitrates, or when the substance examined is sodium or potassium nitrate; but by a slight modification, satisfactory results can be obtained even with nitrates themselves.

It is of great importance that the phenol-sulphonic acid should not be allowed to act upon compact masses of the substance.

One gram of the material is treated in a flash with 25 cc. of water and evaporated to dryness in an air bath at 100–110° C. This distributes the nitrates evenly throughout the mass. After cooling, 50 cc. of sulphuric acid containing 20 grams of phenol per liter is added. This converts the nitrates into nitro-phenol. After standing for a few minutes, 2 to 3 grams of zinc dust and 1 or 2 drops of metallic mercury are added and the mixture boiled for about one and one-half hours. This converts the nitro-

phenol into amido-phenol, and then into sulphate of ammonia. The process is then completed in the usual way.

The writer made a number of tests with this method on the samples sent out by the committee and on pure potassium nitrate, with the following results:

	No. 4.	No. 5.	No. 6.	Pot. nit. c. p.
No. 1	3.24	3.06	2.57	13.74
No. 2	3.30	2.97	2.59	13.81
No. 3	3.27	3.03	2.63	13.79
Mean	3.27	3.02	2.59	13.78
Theory	3.28	3.04	2.58	13.84

These results show that when this method is carefully carried out it gives very accurate results, with fertilizers containing nitrates and with pure salts.

Professor Scovell of this committee, in a letter to the writer, describes the following modifications of the Jodlbaur-Kjeldahl method: .7 gram of potassium nitrate was placed in a Kjeldahl flash, 30 cc. of sulphuric acid added; then 3 cc. of phenol-sulphonic acid (50 grams phenol to 100 cc. sulphuric acid), and 3 grams of zinc dust and 2 drops of platinic chloride solution. After the mixture began boiling and fumes ceased 0.7 gram of mercuric oxide were added and the operation finished as usual.

The following results were obtained by Professor Scovell, on committee's samples and pure potassium nitrate:

	No. 4.	No. 5.	No. 6.	Potassium nitrate, c. p.
No. 1	3.30	2.94	2.49	13.70
No. 2	3.10	2.92	2.49	13.75
No. 3	....	2.92	2.52	.....
Mean	3.20	2.93	2.50	13.73
Theory	3.28	3.04	2.58	13.84

Care should be taken that the reaction is not too great. When lower oxides of nitrogen are present some escape. To avoid this loss the following modification was tried with good results. Salicylic acid method: 0.7 gram of substance is put in a Kjeldahl flash; then 30 cc. sulphuric acid containing 2 grams of salicylic acid, then zinc dust, &c., as usual.

The following results were obtained by Professor Scovell and the writer on the above modification, except that the writer first dissolved in water as recommended by Stutzer and Reitmaier (Rep. Anal. Chem., **7**, 4-6):

	Number 4.	Number 5.	Number 6.	Potassium nitrate, c. p.
M. A. Scovell	3.21	2.97	2.55	13.60
	3.32	2.99	2.61	13.78
	.....	3.05	.....	13.85
	.....	.....	.....	13.82
	.....	.....	.....	13.83
	.....	.....	.....	13.82
	.....	.....	.....	13.88
Mean	3.27	3.00	2.58	13.80
W. J. Gascoyne	3.23	2.95	2.56	13.73
	3.33	3.04	2.52	13.82
	3.25	3.00	2.60	13.80
Mean	3.27	2.99	2.56	13.78
Theory	3.28	3.04	2.58	13.84

From the results obtained by the above method, we believe that it is applicable for the estimation of nitrogen in all forms.

In the annual report of the New Jersey Agricultural Experiment Station for 1886 is published a paper by Dr. A. T. Neale on "The examination and comparison of analytical methods for the determination of nitrogen." In reference to the Kjeldahl method, he says:

"No difficulty was experienced in oxidizing the most refractory substance in from one to three hours. When care was taken to continue the digestion until a perfectly colorless solution was obtained, the addition of potassium permanganate did not appear necessary; its absence also seemed to lessen the liability of the solution to bump."

When caustic soda was used the boiling was perfectly smooth; with caustic potash, however, the bumping of the solution is so serious as to make it difficult to finish the determination. A comparison of the results (between the soda-lime and Kjeldahl methods) warrants the following conclusions:

The average determinations of one hundred and twenty samples of complete fertilizers show that higher percentages of nitrogen were secured by the Kjeldahl process; the average difference for the whole number of samples between the results from these two methods is five hundredths of one per cent. Of the twenty-five samples of high grade organic nitrogenous material analyzed the average percentage of nitrogen is identical by both methods.

In ground bones, dissolved bones, and miscellaneous materials the average determinations of twenty-seven samples by Kjeldahl's method were seven-hundredths of one per cent. lower than by the soda-lime process.

The Kjeldahl method secured slightly higher percentages of nitrogen in coarse fodder samples.

M. Raulin (Bull. Soc. Chimique, **47**, No. 2, page 92; Chem. News, **55**, 147) describes a modification of Dumas's method for the determination of total nitrogen in organic matter. The method consists of an apparatus for generating carbonic acid, on Deville's continuous system, from marble and hydrochloric acid. This apparatus transmits a current of carbonic acic (regulated by pinchcocks) through the combustion-tube. This is a tube of copper, 180 cm. in length and 18 mm. in diameter, surrounded by four brass jackets. Each of these jackets is 10 cm. in length, and is fitted with two tubulures by which the jackets communicate with each other through caoutchouc tubes. Two of these jackets are at the ends of the combustion-tube, whilst the two others are placed equidistant in the intervening space. During the combustion a current of cold water passes through the jackets to cool the central tube. This central tube communicates with an apparatus for collecting the gas.

The apparatus is used as follows: A spiral cylinder of copper wire gauze about 6 cm. long is introduced into the tube, so that the anterior extremity will be 10 cm. from the anterior jacket, then by means of a long dry glass funnel introduce 60 grams oxide of copper; then the mixture of the organic matter and oxide of copper. It is first mixed with a little copper oxide in fine powder and 20 grams of the oxide in grains to .8 gram of the material; the funnel is then rinsed out with a little oxide in grains.

In the same manner a sample of another organic substance is introduced in the second interval of the tube between the second and third jacket, with a second cylinder of copper gauze and oxide. A third sample is introduced into the third interval. The tube is then placed in the furnace, a current of carbonic acid is passed in to clear the tube, and when the residue of the gas not absorbable by caustic alkali which enters the graduated tube does not exceed $\frac{1}{2}$ cc. in a quarter of an hour a current of water is passed through the jacket. The combustion of the first sample is then started working from the front backwards. When the gas no longer continues to increase the volume of gas is read off. The combustion of the second and third samples is then proceeded

with in the same manner. A formula is given for the calculation of the percentage of nitrogen.

RESULTS OF WORK DONE DURING THE YEAR.

Last October your committee sent to nineteen official and nine commercial chemists six samples for nitrogen determination.

Samples No. 1–3 prepared for the purpose of comparing the soda-lime and Kjeldahl methods, and samples No. 4–6 for the purpose of thoroughly testing the Ruffle and copper-oxide methods.

In sample No. 6, members were requested to determine the nitrogen present as ammonia, as nitrates, and as organic nitrogen.

The following is a description of the samples:

No. 1. Dried blood, 13.82 per cent. nitrogen.

No. 2. Acid phosphate and dried blood, containing 2.76 per cent. of nitrogen.

No. 3. Acid phosphate, dried blood, and cotton-seed meal, containing 3.30 per cent. of nitrogen.

No. 4. Acid phosphate and c. p. nitrate of soda, containing 3.28 per cent. of nitrogen.

No. 5. Acid phosphate, dried blood, and c. p. nitrate of soda, containing 3.04 per cent. of nitrogen.

No. 6. Acid phosphate, dried blood, c. p. nitrate of soda, and c. p. sulphate of ammonia, containing 2.58 per cent. of nitrogen. No. 6 contains 1.06 per cent. of nitrogen from ammonia salts, .82 per cent. from nitrates, and .70 per cent. from organic material.

We regret to say that only eight official and two commercial chemists have reported on the samples.

The results obtained are given in the following tables:

SODA-LIME METHOD.

	No. 1.	No. 2.	No. 3.
A	13.50	2.85	3.09
C	14.20	2.80	3.35
D	14.23	3.00	3.13
E	13.49	2.97	3.08
H	13.85	2.80	3.29
I	13.80	3.17	3.30
K	13.44	2.73	3.01
Average	13.79	2.90	3.18

KJELDAHL METHOD.

A	13.44	2.87	3.11
B	13.95	2.74	3.37
C	14.03	2.48	3.36
E	13.86	2.82	3.27
F	14.04	2.83	3.30
G		2.87	3.21
H	13.90	2.85	3.26
I	13.86	2.85	3.25
K	13.46	2.73	3.22
Average	13.82	2.79	3.26

RUFFLE METHOD.

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
A	13.40	2.85	3.09	3.30	3.12	2.60
B				3.08	2.91	2.53
C	14.22	3.07	3.64	3.39	2.82	2.50
D				3.04	2.81	2.54
E	13.83	3.24	3.16	2.70	2.91	2.57
G						2.55
H	13.96	2.82	3.29	3.32	2.97	2.59
I	13.88	2.81	3.28	2.92	2.69	2.26
K	13.37	2.77	3.19	2.31	2.77	2.45
Average	13.78	2.92	3.26	3.01	2.87	2.51

COPPER-OXIDE METHOD.

B				3.28	3.04	2.59
F				3.30	3.02	2.59
H	13.92	2.87	3.30	3.29	3.02	2.57
Average	13.92	2.87	3.30	3.29	3.03	2.58

METHODS OTHER THAN OFFICIAL

A	13.50	2.90	3.10	3.40	3.15	2.60
E	13.44	2.57	3.05	3.30	3.14	2.52
F				3.38	3.04	2.61
H^1				3.30	3.04	2.59
H^2				3.27	3.02	2.59
H^3				3.27	2.99	2.56
I				3.27	3.00	2.58
Average	13.47	2.73	3.08	3.31	3.05	2.58

METHODS USED.

A. Original Ruffle.
E. Original Ruffle, substituting sugar for the c. and s. mixture.
H^1. Official Ruffle, substituting sugar for the c. and s. mixture.
H^2. Stutzer & Reitmaier's modification of the Kjeldahl method.
H^3 and I. Scovell's salicylic acid method.
F. Kjeldahl using 1 gram of substance, 1 gram benzoic acid, 1 gram metallic mercury, 2½ grams zinc and 25 cc. sulphuric acid. In other respects the treatment was similar to the ordinary method.

Nitrogen in sample No. 6.

	Nitrogen as ammonia.	Nitrogen as nitrates.	Organic nitrogen.	Total nitrogen.
A	1.02	.83	.72	2.57
G	1.02	.69	.85	2.55
H	1.04	.80	.72	2.56
Average	1.03	.77	.76	2.56

In obtaining these results A and H used the following method: One gram of the sample was exhausted with water and filtered. In the filtrate nitrogen from ammonia salts was determined by distillation with calcined magnesian oxide, and nitrogen from nitrates in the same fluid, by distillation with caustic potash and zinc and iron filings. The exhausted residue was dried and the nitrogen in organic matter determined by the soda-lime method.

Averages.

SODA-LIME METHOD.

Sample.	Number of determinations.	Average.	Highest.	Lowest.	Difference.
No. 1	7	13.79	14.23	13.44	.79
No. 2	7	2.90	3.17	2.80	.37
No. 3	7	3.18	3.35	3.01	.34

KJELDAHL METHOD.

No. 1	7	13.81	14.04	13.44	.60
No. 2	8	2.77	2.87	2.48	.39
No. 3	8	3.26	3.37	3.11	.26
No. 4	4	3.29	3.38	3.27	.11
No. 5	4	3.01	3.04	2.99	.05
No. 6	4	2.58	2.61	2.56	.05

RUFFLE METHOD.

No. 1	5	13.78	14.28	13.40	.88
No. 2	5	2.92	3.24	2.81	.33
No. 3	5	3.28	3.64	3.09	.55
No. 4	7	3.01	3.39	2.70	.69
No. 5	7	2.87	3.12	2.69	.43
No. 6	8	2.51	2.60	2.26	.34

COPPER-OXIDE METHOD.

No. 4	3	3.29	3.30	3.28	.02
No. 5	3	3.03	3.04	3.02	.02
No. 6	3	2.58	2.59	2.57	.02

Nitrogen in sample No. 6.

	Number of determinations.	Average.	Highest.	Lowest.	Difference.
Nitrogen as ammonia	3	1.03	1.04	1.02	.02
Nitrogen as nitrates	3	.77	.83	.69	.14
Organic nitrogen	3	.76	.85	.71	.13
Total nitrogen	3	2.53	2.57	2.55	.02

In connection with these results, the following points may be mentioned:

The averages agree reasonably well with the calculated results, but the differences between the determinations are too great to pass unnoticed.

By the soda-lime method the average of sample No. 1 is .03 lower than the mean of all the determinations by the official methods on this sample.

On sample No. 2 the average is .14 higher than theoretical results.

On sample No. 3 the average is .12 lower than calculated results.

By the Kjeldahl method, the average of sample No. 1 is identical with the mean of all tests on this sample.

On sample No. 2 the average is .03 higher than theoretical.

On sample No. 3 the average is .02 lower than theoretical.

On samples Nos. 4, 5, and 6, by the modifications of the Kjeldahl method noticed in this report, the results are as follows:

On sample No. 4 the average is .01 higher than theoretical.

On sample No. 5 the average is .03 lower than theoretical, and on sample No. 6 the average and theoretical results are identical.

The results by the Ruffle method are not very satisfactory. Many have obtained theoretical results, while others have reported determinations so much below the calculated results that we are satisfied that it is not altogether the fault of the method.

On sample No. 1 the average is .04 lower than the mean of all determinations.

On sample No. 2 the average is .06 higher than theoretical.

On sample No. 3 the average is .01 lower than theoretical.

On sample No. 4 the average is .27 lower than theoretical. Omitting these very out-of-the-way determinations, the average would only be .05 lower than calculated.

On sample No. 5 the average is .15 lower than theoretical.

On sample No. 6 the average is .07 below theoretical, and by leaving out one determination, which is much too low, the average would be only .04 below theoretical.

The results by the copper-oxide method are very satisfactory.

On sample No. 3 the average is .01 higher than theoretical; sample No. 4 is .01 lower, and on sample No. 6 the average and theoretical results are identical.

On sample No. 6 the following results were obtained: On nitrogen and ammonia the average is .03 below theoretical; on nitrogen as nitrates the average is .05 below, and for organic nitrogen the average is .06 higher than calculated results.

RECOMMENDATIONS.

After the careful consideration of the results obtained, the committee desires to make the following recommendations:

(1) That the copper-oxide method be continued.

(2) That the Ruffle method be continued, except that sugar be substituted for the charcoal and sulphur mixture.

(3) That the present official Kjeldahl and soda-lime methods be used where no nitrates are present.

(4) That while on account of the want of experience of the members of this Association in the use of the salicylic acid modification of the Kjeldahl method as recommended by Prof. Scovell, the committee does not feel authorized to recommend its adoption as an official method, but desire to present it to the careful consideration of the Association, in the belief that its simplicity and adaptability to all the contingencies of the fertilizer analysis are such as to render advisable its adoption and use.

Respectfully submitted,

W. J. GASCOYNE, *Chairman.*
M. A. SCOVELL.

Professor M. A. SCOVELL, a member of the nitrogen committee, then read the following paper:

THE KJELDAHL METHOD APPLICABLE TO DETERMINATION OF NITROGEN IN NITRATES.

In looking over the field for investigation, as a member of your Committee on Nitrogen, it seemed to me the one object to be obtained was some modification of the Kjeldahl method, so as to make it applicable in all cases of nitrogen determinations.

The absolute method as adopted by the Association is all that can be desired so far as quantitative accuracy is concerned. But the great length of time it takes to estimate nitrogen by this process is a cause for looking for a simpler and more rapid method.

The Ruffle method, as adopted by the Association, needs at least to be perfected. In my hands the results have been discouraging. Especially is this so where pure nitrates

are examined. In estimating pure KNO_3 by the method as adopted by the Association, the following results were obtained:

	Nitrogen.
	Per cent.
1	11.12
2	12.56
3	12.04
4	11.68

A modification, whereby sugar was used in place of charcoal, gave 12.88. Theory, 13.83.

Where small quantities of nitrates are present this method works better. But it seems to me a method that will not give satisfactory results on pure nitrates is to be used with caution. Furthermore, the manipulations required and the time consumed in the determinations, like that of the absolute method, make a more rapid method desirable.

As to rapidity of execution the Kjeldahl method is all that can be desired.

The benzoic-acid method has been investigated by Mr. Spencer, of the Department of Agriculture, with very unsatisfactory results (see Bulletin No. 12). M. Jodlbaur (Chem. Centr., 1886, 433) also finds the results by this method far too low; but he made a series of experiments with phenol-sulphuric acid and obtained satisfactory results. The method is given in the committee's report. Stutzer and Reitmaier claim that Jodlbaur's method does not give accurate results when the manure contains large quantities of nitrates or when $NaNO_3$ or KNO_3 are examined, and suggest adding water to the fertilizer, evaporating in the Kjeldahl flask, and then heating with sulphuric acid containing phenol. This, they claim, distributes the nitrates evenly throughout the mass.

In our hands M. Jodlbaur's method on pure potassium nitrate gave the following results:

	Per cent.
Nitrogen	13.70
	13.75
	13.74
Average	13.73
Theoretical	13.84

The average is 0.11 per cent. below the theoretical. Not all that could be desired, yet nearly approaching satisfactory results.

We next tried this method on sodium nitrate. For this purpose samples Nos. 4, 5, and 6 of the nitrogen committee were selected.

Sample No. 4 is a mixture of acid phosphate and sodium nitrate, containing 3.28 per cent. nitrogen.

	Per cent.
Nitrogen found	3.10
	3.30
Average	3.20

Sample No. 5, acid phosphate, blood and pure sodium nitrate, containing 3.04 per cent. nitrogen.

	Per cent.
Nitrogen found	2.94
	2.92
	2.92
Average	2.93

Sample No. 6, acid phosphate, blood, sulphate of ammonia, and pure sodium nitrate, containing 2.58 per cent. nitrogen.

	Per cent.
Nitrogen found	2.49
	2.49
	2.52
Average	2.50

Being convinced that the loss of nitrogen was due to its escape when the phenol mixture was added to the nitrate, I endeavored to overcome this difficulty without having to resort to the tedious process of dissolving the nitrates in water, evaporating, and spreading over the flask as Stutzer and Reitmaier recommend.

Salicylic acid dissolves readily in sulphuric acid without rise of temperature or vigorous reaction.

Taking advantage of this we continued our experiments, substituting salicylic acid for phenol-sulphuric acid, and also adding 0.7 grams of mercuric oxide after digestion for sometime to hasten oxidation.

Our first experiment was on pure potassium nitrate. This contained .07 per cent. water, theoretical per cent. nitrogen 13.83. 0.7 grams of potassium nitrate was brought into a Kjeldahl flask, and dissolved in 30 cc. sulphuric acid. Two grams salicylic acid were added, and contents shaken; then 3 grams zinc-dust gradually added and 2 drops of a platinic chloride solution. The whole was first gradually heated, until danger of frothing was over, then the temperature increased until the liquid boiled rapidly in the flask. After vigorous reaction ceased, 0.7 grams mercuric oxide were added and the boiling continued until the liquid was colorless. This generally took about one hour from the time digestion first begun. In the distillation the official Kjeldahl method was followed.

By this method the following results were obtained on the potassium nitrate:

	Per cent.
Nitrogen found	13.60
	13.78
	13.85
	13.82
	13.83
	13.83
	13.82
	13.88
Average	13.80
Theoretical	13.83

Committee samples Nos. 4, 5, and 6, containing sodium nitrate, and, as described before, were analyzed by this method with the following results:

	Sample No. 4.	Sample No. 5.	Sample No. 6.
	Per cent.	*Per cent.*	*Per cent.*
Nitrogen found	3.21	2.97	2.55
	3.32	2.99	.....
	.....	3.05	2.61
Average	3.27	3.00	2.58
Theoretical	3.28	3.04	2.58

The next experiment was the determination of nitrogen in a sample of pure $NaNO_3$, containing sugar:

	Per cent.
Nitrogen found	3.37
	3.38
Nitrogen found by the absolute method	3.38

In the selection of samples to test the method due regard was taken to have the nitrogen in all forms, as found in fertilizers, after testing the method first by pure potassium nitrate. Samples Nos. 4, 5, and 6 were mixed last September, almost a year ago, and the sample with sugar over one year. This certainly would give the nitrogen in a form more nearly representing the nitrogen fertilizer mixtures than samples freshly mixed. In sample No. 6 all three of the forms of nitrogen were represented. The method seems to give very accurate results, whether pure salts are under examination or the various mixtures representing fertilizer mixtures.

For ease of manipulation and rapidity of work this method especially recommends itself, and I believe it will prove to be satisfactory in every respect.

Dr. WILEY then read the following:

ABSTRACT OF THE REPORT OF THE COMMITTEE ON THE DETERMINATION OF NITROGEN IN FERTILIZERS, PRESENTED TO THE FRENCH ASSOCIATION OF SUGAR CHEMISTS, JULY, 1887.

The committee consisted of Messrs. Deherain, Julie, Aubin, and Dupont.

NITROGEN.

A preliminary examination was recommended to determine the nature of the nitrogen in a sample.

AMMONIA SALTS.

A small portion is mixed with powdered soda lime in the cold. If ammonia salts are present the odor of ammonia is observed.

NITRIC ACID.

Nitric acid is detected by sulphuric acid and ferrous sulphate in the aqueous extract of the sample. If the aqueous solution be too highly colored, alcohol of 80 per cent. strength should be employed as the solvent.

Brucine may also be used to detect the nitric acid. Mix 1cc. aqueous solution of brucine with the same quantity of the solution of the fertilizer; add carefully 1 cc. strong sulphuric acid, letting it run slowly down the side of the test tube. If nitric acid be present a rose color varying to yellow and very persistent will be produced.

ORGANIC NITROGEN.

Nitrogen existing in organic compounds may be detected by heating the residue from the aqueous extraction with soda lime.

QUANTITATIVE ESTIMATION OF NITROGEN.

The committee divide the process into eight parts, viz.:

(1) Estimation of organic nitrogen.
(2) Estimation of ammoniacal nitrogen.
(3) Estimation of nitric nitrogen.
(4) Procedure in case of a fertilizer containing nitrogen in its three forms.
(5) Procedure in case of a fertilizer containing organic and ammoniacal nitrogen.
(6) Procedure in case of a fertilizer containing organic and nitric nitrogen.
(7) Procedure in case of a fertilizer containing ammoniacal and nitric nitrogen.
(8) Estimation of total nitrogen.

QUANTITATIVE ESTIMATION OF ORGANIC NITROGEN.

Combustion with soda lime and the Kjeldahl method are recommended. The standard acid is recommended to be of such a strength that 1 cc. may correspond to one-hundredth of a gram of nitrogen. For this purpose 35 grams of monohydric sulphuric acid or 45 grams of crystallized oxalic acid are required to the liter.

QUANTITATIVE ESTIMATION OF AMMONIACAL NITROGEN.

The process of Boussingault is recommended, namely: the evolution of the ammonia by means of lime, potash, or magnesia.

MANIPULATION.

In a flask of 500 cc. capacity is placed half a gram of the material to be analyzed, together with 30 cc. of distilled water and 2 grams of caustic magnesia. This flask is connected with the condensing apparatus and the distillation is continued until 60 to 100 cc. have passed over. The titration is made with standard alkali, using litmus or lacmoïd as an indicator. The distillation apparatus of Aubin is recommended, in which the distillation is made *per ascensum*, the greater part of the vapor of water being condensed and returned into the flask, while the ammonia, more volatile, escapes in such a way as to secure the whole of the ammonia in a very small portion of the water. This renders the titration more exact.

QUANTITATIVE ESTIMATION OF NITRIC NITROGEN.

The process of Schloesing is recommended. It rests upon the transformation of nitric acid into dioxide of nitrogen.

The reaction which takes place is as follows:

$$6FeCl_2 + 2NO_3MO + 8HCl = 2MCl + 4H_2O + 2NO_2 + 3Fe_2Cl_6$$

The dioxide of nitrogen which is set free is measured and from its volume the weight of nitrogen is calculated. When the matter to be analyzed is not very pure, or when it is mixed with a soluble carbonate, it is possible that other gases beside the dioxide of nitrogen may be set free. In this case it is necessary that a correction to the total volume should be made.

When the operation is finished the dioxide of nitrogen can be absorbed by introducing into the tube containing it a piece of ferrous sulphate. The tube should be shaken sufficiently long and vigorously to insure the complete absorption of the dioxide of nitrogen. The reading of the gaseous volume before and after the introduction of the ferrous sulphate will give the volume of gas corresponding to the dioxide of nitrogen.

Further detailed directions for conducting the operation and calculating the weight of nitrogen are given in the report of the committee.

ESTIMATION OF NITROGEN IN ITS THREE STATES IN A COMPLEX FERTILIZER.

1. Weigh 10 grams of the matter, place in a flask containing water, shake, and allow to dissolve, filter and wash until the volume of the wash water amounts to 500 cc.

(*a*) Take 50 cc. of the liquid and treat it for ammonia by Boussingault's method.

(*b*) On another part of the liquid, estimate the nitric nitrogen by Schloesing's process.

2. A new sample of the fertilizer is taken and the ammoniacal and nitric nitrogen which it contains destroyed, as follows:

The ammonia is driven off by boiling the sample in a porcelain dish with caustic magnesia and a sufficient quantity of water. Afterwards evaporate to dryness, cover the sample with some oxalic acid, and moisten. Dry in an oven at 110° for an hour and a half. All of the nitric nitrogen is transferred into dioxide of nitrogen, which escapes. The organic nitrogen which remains is estimated by soda-lime or by the Kjeldahl process.

FERTILIZER CONTAINING ORGANIC NITROGEN AND NITRIC NITROGEN.

1. The nitric nitrogen is estimated in the aqueous solution by the method of Schloesing.

2. From 1 to 2 grams of the substance the nitric acid is driven off by oxalic acid in the manner described above, and the organic nitrogen estimated in the residue by means of the soda-lime or Kjeldahl method.

FERTILIZER CONTAINING ORGANIC NITROGEN AND AMMONIACAL NITROGEN.

1. The ammoniacal nitrogen is determined by the method of Boussingault.

2. For the determination of the organic nitrogen 1 to 2 grams of the fertilizer are boiled in a porcelain dish with magnesia and a sufficient quantity of water to drive off the ammonia. Evaporate to dryness and estimate the organic nitrogen by the soda-lime or Kjeldahl process.

FERTILIZER CONTAINING AMMONIACAL NITROGEN AND NITRIC NITROGEN.

Weigh out 10, 20, or 30 grams, and add enough water to make up to 500 cc. Take 50 or 100 cc. and estimate the ammoniacal nitrogen in the manner already described. On another portion of the liquid the nitric nitrogen is estimated by the method of Schloesing.

ESTIMATION OF TOTAL NITROGEN.

The absolute method is recommended. Two methods of procedure are indicated:

(1) The apparatus may be freed of air by means of carbonic dioxide, or (2) the air may be exhausted by means of a mercury pump.

The committee gives preference to the latter procedure.

Full details of the manipulation are given by the committee.

The report of the Committee on Nitrogen being open for discussion, the Kjeldahl method was taken up.

Professor SCOVELL described his results in determining nitrates by the use of phenol and salicylic acid, and Dr. GASCOYNE said that he had been able to confirm them.

Mr. FARRINGTON then described his method with phenol, as follows:

THE JODLBAUR MODIFICATION OF THE KJELDAHL METHOD [CHEM. ZTG., 1887 2, 12] WHEN NITRATES ARE PRESENT.

To 1 gram substance in a dry, pear-shaped flask of about 250 cc. capacity, add 50 cc. of a solution containing 50 grams phenol in 1,000 cc. common sulphuric acid.

After standing some minutes, with frequent shaking, add 2 to 3 grams zinc-dust and about 7 grams mercury monoxide.

Heat the flask at first over a low flame, as its contents may foam badly; finally increase the heat till the acid liquid in the flask boils vigorously.

Continue the boiling till the solution is nearly colorless, then add potassium permanganate and proceed as in the usual Kjeldahl method for determining nitrogen.

I use common sulphuric acid, as there is less danger of driving off nitric acid when it is added to a nitrate than by using c. p. sulphuric acid.

The common sulphuric acid also contains less nitrogen than any c. p. acid that I have been able to get.

I have made a determination of nitrogen by this modification of the Kjeldahl method, comparing it with one made by the absolute or copper-oxide method on forty different samples of commercial fertilizers, containing total nitrogen from 2½ to 6½ per cent., of which from ½ to 2½ per cent. was in the form of nitrates.

The difference in results by the two methods was in 55 per cent. of the analyses less than .1 per cent.; in 24 per cent. of the analysis, between .1 and .15 per cent.; in 14 per cent. of the analysis, between .15 and .20 per cent.; in 7 per cent. of the analysis, between .20 and .24 per cent.

Professor SCOVELL said he found less nitrogen with phenol than with salicylic acid.

Mr. CHAZAL then moved that Professor Scovell's method be adopted as an addition to the Kjeldahl method, and it was adopted.

The absolute method was also sanctioned for another year, and, after discussion, the Ruffle method substituting sugar for charcoal and soda lime for slaked lime.

The soda-lime method was also retained as being necessary for some chemists not having facilities for working in other ways.

The report, with amendments, was then recommitted and, on its return by the committee, adopted as a whole.

Miscellaneous business being in order, the committee on amendments to the constitution reported a recommendation that the second section should read as follows:

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership, and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to a vote in the Association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the Association by presenting proper credentials and signing the constitution. All analytical chemists and others interested in the objects of the Association may attend its meetings and take part in its discussions, but shall have no vote in the Association.

The recommendation was accepted and unanimously adopted.

Professor MYERS then moved to refer the method of electing officers to a committee to report at next meeting, which was carried.

Professors Myers, Stubbs, and Gascoyne were appointed on the committee.

Mr. RICHARDSON moved, and it was adopted, that hereafter the names of persons making analyses of the official samples be attached to their results on publication.

Professor RISING moved that a committee be appointed on wines or fermented liquors; carried.

Professor STUBBS moved that a committee be appointed on sugar and sugar products; carried.

The committee to recommend officers then reported the following list, which was unanimously elected:

For President: Mr. P. E. Chazal.

For Vice-President: Dr. W. J. Gascoyne.

For Secretary: Mr. Clifford Richardson.

For Executive Committee: Dr. E. H. Jenkins, Prof. J. A. Myers.

Professor STUBBS then moved and it was unanimously carried—

That the thanks of the Association are tendered to the honorable Commissioner of Agriculture for his kindness to and interest in the Association, and for his continued generosity in publishing and distributing its proceedings, which have largely aided in extending its influence.

The convention then adjourned *sine die* to await the call of the Executive Committee.

Appendix A.

OFFICIAL METHODS OF ANALYSIS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR 1887-'88.

METHODS FOR DETERMINING PHOSPHORIC ACID AND MOISTURE.

(1) *Preparation of sample.*—The sample should be well intermixed and properly prepared, so that separate portions shall accurately represent the substance under examination, without loss or gain of moisture.

(2) *Determination of moisture.*—(*a*) In potash salts, nitrate of soda, and sulphate of ammonia heat 1 to 5 grams at 130° C. till the weight is constant, and reckon water from the loss. (*b*) In all other fertilizers heat 2 grams, or if the sample is too coarse to secure uniform lots of 2 grams each, 5 grams, for five hours at 100° in a steam bath.

(3) *Water-soluble phosphoric acid.*—Bring 2 grams on a filter, add a little water, let it run out before adding more water, and repeat this treatment cautiously until no phosphate is likely to precipitate in the filter. If the washings show turbidity after passing the filter clear up with acid. When the substance is nearly washed in this manner it is transferred to a mortar and rubbed with a rubber-tipped pestle to a homogeneous paste (but not further pulverized), then returned to the filter and washed with water until the filtrate measures not less than 250 cc. Mix the washings. Take an aliquot (usually corresponding to $\frac{1}{5}$ or $\frac{1}{2}$ gram of the substance) and determine phosphoric acid, as under total phosphoric acid.

(4) *Citrate-insoluble phosphoric acid.*—Wash the residue of the treatment with water into a 200 cc. flask with 100 cc. of strictly neutral ammonium citrate solution of 1.09 density, prepared as hereafter directed. Cork the flask securely and place it in a water bath, the water of which stands at 65° C. (The water bath should be of such a size that the introduction of the cold flask or flasks shall not cause a reduction of the temperature of the bath of more than 2° C.) Raising the temperature as rapidly as practicable to 65° C., which is subsequently maintained, digest with frequent shakings for thirty minutes from the instant of insertion, filter the warm solution quickly (best with filter-pump), and wash with water of ordinary temperature. Transfer the filter and its contents to a capsule, ignite until the organic matter is destroyed, treat with 10 to 15 cc. of concentrated hydrochloric or nitric acid, digest over a low flame until the phosphate is dissolved, dilute to 200 cc., mix, pass through a dry filter, take an aliquot and determine phosphoric acid as under total.

In case a determination of citrate-insoluble phosphoric acid is required in non-acidulated goods, it is to be made by treating 2 grams of the phosphatic material, without previous washing with water, precisely in the way above described, except that in case the substance contains much animal matter (bone, fish, &c.) the residue insoluble in ammonium citrate is to be treated by one of the processes described below.

(5) *Total phosphoric acid.*—Weigh 2 grams and treat by one of the following methods: (1) Evaporation with 5 cc. magnesium nitrate, ignition, and solution in acid. (2) Solution in 30 cc. concentrated nitric acid with a small quantity of hydrochloric acid.

(3) Add 30 cc. concentrated hydrochloric acid, heat, and add cautiously and in small quantities at a time about 0.5 gram of finely pulverized potassium chlorate.

Boil gently until all phosphates are dissolved and all organic matter destroyed: dilute to 200 cc.; mix and pass through a dry filter; take 50 cc. of filtrate; neutralize with ammonia (in case hydrochloric acid has been used as a solvent add about 15 grams dry ammonium nitrate or its equivalent). To the hot solutions for every decigram of P_2O_5 that is present, add 50 cc. of molybdic solution. Digest at about 65° C. for one hour, filter, and wash with ammonium nitrate solution. (Test the filtrate by renewed digestion and addition of more molybdic solution.) Dissolve the precipitate on the filter with ammonia and hot water and wash into a beaker to a bulk of not more than 100 cc. Nearly neutralize with hydrochloric acid, cool, and add magnesia mixture from a burette; add slowly (one drop per second), stirring vigorously. After fifteen minutes add 30 cc. of ammonia solution of density 0.96. Let stand several hours (two hours is usually enough). Filter, wash with dilute ammonia, ignite intensely for ten minutes, and weigh.

(6) Citrate-soluble phosphoric acid. The sum of the water-soluble and citric insoluble subtracted from the total gives the citrate-soluble.

PREPARATION OF REAGENTS.

(1) *To prepare ammonium citrate solution.*—Mix 370 grams of commercial citric acid with 1,500 cc. of water; nearly neutralize with crushed commercial carbonate of ammonia; heat to expel the carbonic acid; cool; add ammonia until exactly neutral (testing by saturated alcoholic solution of coralline) and bring to volume of two liters. Test the specific gravity, which should be 1.09 at 20° C., before using.

(2) *To prepare molybdic solution.*—Dissolve 100 grams of molybdic acid in 400 grams or 4 7 cc. of ammonia of specific gravity 0.96, and pour the solution thus obtained into 1,500 grams or 1,250 cc. of nitric acid of specific gravity 1.20. Keep the mixture in a warm place for several days, or until a portion heated to 40° C. deposits no yellow precipitate of ammonium phospho-molybdate. Decant the solution from any sediment, and preserve in glass-stoppered vessels.

(3) *To prepare ammonium nitrate solution.*—Dissolve 200 grams of commercial ammonium nitrate in water and bring to a volume of two liters.

(4) *To prepare magnesia mixture.*—Dissolve 22 grams of recently ignited calcined magnesia in dilute hydrochloric acid, avoiding excess of the latter. Add a little calcined magnesia in excess, and boil a few minutes to precipitate iron, alumina, and phosphoric acid, filter, add 280 grams of ammonium chloride, 700 cc. of ammonia of specific gravity 0.96, and water enough to make the volume of two liters. Instead of the solution of 22 grams of calcined magnesia 110 grams of crystallized magnesium chloride ($MgCl_2, 6H_2O$) may be used.

(5) *Dilute ammonia for washing.*—One volume ammonia of specific gravity 0.96 mixed with three volumes of water, or usually one volume of concentrated ammonia with six volumes of water.

(6) *Nitrate of magnesia.*—Dissolve 320 grams of calcined magnesia in nitric acid, avoiding an excess of the latter; then add a little calcined magnesia in excess, boil; filter from excess of magnesia, ferric oxide, &c., and bring to volume of two liters.

METHODS OF DETERMINING POTASH.

METHOD OF LINDO AS MODIFIED BY GLADDING.

(1) *Superphosphates.*—Boil 10 grams of the fertilizer with 300 cc. of water for ten minutes. Cool the solution; add ammonia in slight excess, thus precipitating all phosphate of lime, oxide of iron, and alumina, &c., make up to 500 cc., mix thoroughly and filter through a dry filter; take 50 cc. corresponding to 1 gram, evaporate nearly to dryness, add 1 cc. of dilute H_2SO_4 (1 to 1), and evaporate to dryness and ignite to whiteness. As all the potash is in form of sulphate, no loss need be apprehended by

volatilization of potash, and a full red heat must be used until the residue is perfectly white. This residue is dissolved in hot water plus a few drops of HCl; 5 cc. of a solution of pure NaCl (containing 20 grams NaCl to the liter) and an excess of platinum solution (4 cc.) are now added, and the whole evaporated as usual. The precipitate is washed thoroughly with alcohol by decantation and on filter, as usual. The washing should be continued even after the filtrate is colorless. Ten cc. of the NH_4Cl solution prepared as above are now run through the filter. These 10 cc. will contain the bulk of the impurities, and are thrown away. A fresh portion of 10 cc. NH_4Cl is now run through the filter several times (five or six). The filter is then washed thoroughly with pure alcohol, dried, and weighed as usual. The platinum solution used contains 1 gram metallic plantinum in every 10 cc.

(2) *Muriates of potash.*—In the analysis of these salts an aliquot portion, containing .500 gram, is evaporated with 10 cc. platinum solution plus a few drops of HCl, and washed as before.

(3) *Sulphate of potash, kainite, &c.*—In the analysis of these salts an aliquot portion containing .500 gram is taken, .250 gram of NaCl added, plus a few drops of HCl, and the whole evaporated with 15 cc. platinum solution. In this case special care must be taken, in the washing with alcohol, to remove all the double chloride of platinum and sodium. The washing should be continued for some time after the filtrate is colorless. Twenty-five cubic centimetes of the NH_4Cl solution are employed, instead of 10 cc., and the 25 cc. poured through at least six times to remove all sulphates and chlorides. Wash finally with alcohol, dry, and weigh as usual.

To prepare the washing solution of NH_4Cl, place in a bottle 500 cc. H_2O, 100 grams of NH_4Cl; shake till dissolved. Now pulverize 5 or 10 grams of K_2PtCl_6, put in the bottle, and shake at intervals for six or eight hours; let settle over night; then filter off liquid into a second bottle. The first bottle is then ready for a preparation of a fresh supply when needed.

ALTERNATE METHOD.

Pulverize the fertilizer (200 or 300 grams) in a mortar; take 10 grams, boil for ten minutes with 200 cc. water, and after cooling, and without filtering, make up to 1,000 cc., and filter through a dry paper. In this method, in case the potash is contained in organic compounds, like tobacco stems, cotton-seed hulls, &c., the substance is to be saturated with strong sulphuric acid and ignited in a muffle to destroy organic matter. If the sample have 10 to 15 per cent. K_2O (kainite), take 50 cc. of the filtrate; if from 2 to 3 per cent. K_2O (ordinary potash fertilizers), take 100 cc. of the filtrate. In each case make the volume up to 150 cc., heat to 100°, and add, drop by drop, with constant stirring, slight excess of barium chloride; without filtering, in the same manner, add barium hydrate in slight excess. Heat, filter, and wash until precipitate is free of chlorides. Add to filtrate 1 cc. strong ammonium hydrate, and then a saturated solution of ammonium carbonate until excess of barium is precipitated. Heat. Add now, in fine powder, 0.5 gram pure oxalic acid or 0.75 gram ammonium oxalate. Filter, wash free of chlorides, evaporate filtrate to dryness in a platinum dish, and, holding dish with crucible tongs, ignite carefully over the free flame below red heat until all volatile matter is driven off.

The residue is now digested with hot water, filtered through a small filter, and washed with successive small portions of water until the filtrate amounts to 30 cc. or more. To this filtrate, after adding two drops of strong hydrochloric acid, is added, in a porcelain dish, 5 to 10 cc. of a solution of 10 grams of platinic chloride in 100 cc. of water. The mixture is now evaporated on the water-bath to a thick syrup, or further treated with strong alcohol washed by decantation, collected in a Gooch crucible or other form of filter, washed with strong alcohol, afterwards with 5 cc. ether, dried for thirty minutes at 100° C,, and weighed.

It is desirable, if there is an appearance of white foreign matter in the double salt, that it should be washed, according to the previous method, with 10 cc. of the half-

concentrated solution of NH_4Cl, which has been saturated by shaking with K_2PtCl_6, as recommended by Gladding.

The use of the factor 0.3056 for converting K_2PtCl_6 to KCl and 0.19308 for converting to K_2O is continued.

METHODS FOR THE DETERMINATION OF NITROGEN.

The Absolute or Cupric Oxide Method.

The apparatus and reagents needed are as follows:

APPARATUS.

Combustion tube of best hard Bohemian glass, about 26 inches long and one-half inch internal diameter.

Azotometer of at least 100 cubic centimeters capacity, accurately calibrated.

Sprengel mercury air pump.

Small paper scoop, easily made from stiff writing-paper.

REAGENTS.

Cupric oxide (coarse).—Wire form; to be ignited and cooled before using.

Fine cupric oxide.—Prepared by pounding ordinary cupric oxide in mortar.

Metallic copper.—Granulated copper or fine copper gauze reduced and cooled in stream of hydrogen.

Sodium bicarbonate.—Free from organic matter.

Caustic potash solution.—Dissolve commercial stick potash in less than its weight of water so that crystals are deposited on cooling. When absorption of carbonic acid ceases to be prompt solution must be discarded.

LOADING TUBE.

Of ordinary commercial fertilizers take 1 to 2 grams for analysis. In the case of highly nitrogenous substances the amount to be taken must be regulated by the amount of nitrogen estimated to be present. Fill tube as follows: (1) About 2 inches of coarse cupric oxide. (2) Place on the small paper scoop enough of the fine cupric oxide to fill, after having been mixed with the substance to be analyzed, about 4 inches of the tube; pour on this the substance, rinsing watch glass with a little of the fine oxide and mix thoroughly with spatula; pour into tube, rinsing the scoop with a little fine oxide. (3) About 12 inches of coarse cupric oxide. (4) About 3 inches of metallic copper. (5) About 2½ inches of coarse cupric oxide (anterior layer). (6) Small plug of asbestos. (7) Eight-tenths to 1 gram of sodium bicarbonate. (8) Large, loose plug of asbestos; place tube in furnace, leaving about 1 inch of it projecting; connect with pump by rubber stopper smeared with glycerine, taking care to make connection perfectly tight.

OPERATION.

Exhaust air from tube by means of pump. When a vacuum has been obtained, allow flow of mercury to continue, light gas under that part of tube containing metallic copper, anterior layer of cupric oxide (see 5th above) and bicarbonate of soda. As soon as vacuum is destroyed and apparatus filled with carbonic acid gas, shut off the flow of mercury and at once introduce the delivery tube of the pump into the receiving arm of the azotometer and just below the surface of the mercury seal of the azotometer, so that the escaping bubbles will pass into the air and not into the azotometer, thus avoiding the useless saturation of the caustic potash solution.

When the flow of carbonic acid has very nearly or completely ceased, pass the delivery tube down into the receiving arm, so that the bubbles will escape into the azotometer. Light the jets under the 12-inch layer of oxide, heat gently for a few moments to drive out any moisture that may be present, and bring to red heat. Heat gradually mixture of substance and oxide, lighting one jet at a time. Avoid too rapid evolution of bubbles, which should be allowed to escape at rate of about one per second or a little faster.

When the jets under mixture have all been turned on, light jets under layer of oxide at end of tube. When evolution of gas has ceased turn out all the lights except those under the metallic copper and anterior layer of oxide, and allow to cool for a few moments. Exhaust with pump and remove azotometer before flow of mercury is stopped. Break connection of tube with pump, stop flow of mercury and extinguish lights. Allow azotometer to stand for at least an hour or cool with stream of water until permanent volume and temperature are reached.

Adjust accurately the level of the KOH solution in bulb to that in azotometer, note volume of gas, temperature, and height of barometer; make calculations as usual. The labor of calculation may be much diminished by the use of the tables prepared by Messrs. Battle and Dancy, of the North Carolina Experiment Station (Raleigh, N. C.).

The above details are, with some modifications, those given in the report of the Connecticut Station for 1879 (p. 124), which may be consulted for details of apparatus, should such details be desired.

Determination by the Method of Kjeldahl.

Reagents and apparatus.

(1) Hydrochloric acid whose absolute strength has been determined, (a) by precipitating with silver nitrate and weighing the silver chloride, (b) by sodium carbonate, as described in Fresenius's Quantitative Analysis, second American edition, page 680, and (c) by determining the amount neutralized by the distillate from a weighed quantity of pure ammonium chloride boiled with an excess of sodium hydrate.

(2) Standard ammonia whose strength, relative to the acid, has been accurately determined.

(3) "C. P." sulphuric acid, specific gravity 1.83, free from nitrates and also from ammonium sulphate, which is sometimes added in the process of manufacture to destroy oxides of nitrogen.

(4) Mercuric oxide, HgO, prepared in the wet way. That prepared from mercury nitrate cannot safely be used.

(5) Potassium permanganate tolerably finely pulverized.

(6) Granulated zinc.

(7) A solution of 40 grams of commercial potassium sulphide in one liter of water.

(8) A saturated solution of sodium hydrate free from nitrates, which are sometimes added in the process of manufacture to destroy organic matter and improve the color of the product. That of the Greenbank Alkali Company is of good quality.

(9) Solution of cochineal prepared according to Fresenius's Quantitative Analysis, second American edition, page 679.

(10) Burettes should be calibrated in all cases by the user.

(11) Digestion flasks of hard, moderately thick, well-annealed glass. These flasks are about 9 inches long, with a round, pear-shaped bottom, having a maximum diameter of 2½ inches, and tapering out gradually in a long neck, which is three-fourths of an inch in diameter at the narrowest part, and flared a little at the edge. The total capacity is 225 to 250 cc.

(12) Distillation flasks of ordinary shape, 550 cc. capacity, and fitted with a rubber stopper and a bulb tube above to prevent the possibility of sodium hydrate being

carried over mechanically during distillation. This is adjusted to the tube of the condenser by a rubber tube.

(13) A condenser. Several forms have been described, no one of which is equally convenient for all laboratories. The essential thing is that the tube which carries the steam to be condensed shall be of block tin. All kinds of glass are decomposed by steam and ammonia vapor, and will give up alkali enough to impair accuracy. (See Kreussler and Henzold, Ber. Berichte, *XVII*, 34.) The condenser in use in the laboratory of the Connecticut Experiment Station, devised by Professor Johnson, consists of a copper tank supported by a wooden frame, so that its bottom is 11 inches above the work-bench on which it stands. This tank is 16 inches high, 32 inches long, and 3 inches wide from front to back, widening above to 6 inches. It is provided with a water-supply tube which goes to the bottom and a larger overflow pipe above. The block tin condensing tubes, whose external diameter is $\frac{3}{8}$ of an inch, seven in number, enter the tank through holes in the front side of it near the top, above the level of the overflow, and pass down perpendicularly through the tank and out through rubber stoppers tightly fitted into holes in the bottom. They project about $1\frac{1}{2}$ inches below the bottom of the tank, and are connected by short rubber tubes with glass bulb tubes of the usual shape, which dip into glass precipitating beakers. These beakers are $6\frac{1}{2}$ inches high, 3 inches in diameter below, somewhat narrower above, and of about 500 cc. capacity. The titration can be made directly in them. The seven distillation flasks are supported on a sheet-iron shelf attached to the wooden frame that supports the tank in front of the latter. Where each flask is to stand a circular hole is cut, with three projecting lips, which support the wire gauze under the flask, and three other lips which hold the flask in place and prevent its moving laterally out of place while distillation is going on. Below this sheet-iron shelf is a metal tube carrying seven Bunsen burners, each with a stop-cock like those of a gas-combustion furnace. These burners are of larger diameter at the top, which prevents smoking when covered with fine gauze to prevent the flame from striking back.

(14) The stand for holding the digestion flasks consists of a pan of sheet-iron 29 inches long by 8 inches wide, on the front of which is fastened a shelf of sheet-iron as long as the pan, 5 inches wide and 4 inches high. In this are cut six holes $1\frac{5}{8}$ inches in diameter. At the back of the pan is a stout wire running lengthwise of the stand, 8 inches high, with a bend or depression opposite each hole in the shelf. The digestion flask rests with its lower part over a hole in the shelf and its neck in one of the depressions in the wire frame, which holds it securely in position. Heat is supplied by low Bunsen burners below the shelf. With a little care the naked flame can be applied directly to the flask without danger.

THE DETERMINATION.

One gram of the substance to be analyzed is brought into a digestion flask with approximately 0.7 gram of mercuric oxide and 20 cc. of sulphuric acid. The flask is placed on the frame above described in an inclined position and heated below the boiling point of the acid for from 5 to 15 minutes, or until frothing has ceased. The heat is then raised till the acid boils briskly. No further attention is required till the contents of the flask have become a clear liquid, which is colorless or at least has only a very pale straw color. The flask is then removed from the frame, held upright, and, while still hot, potassium permanganate is dropped in carefully and in small quantity at a time till after shaking the liquid remains of a green or purple color. After cooling, the contents of the flask are transferred to the distilling flask with water, and to this 25 cc. of potassium sulphide solution are added, 50 cc. of the soda solution, or sufficient to make the reaction strongly alkaline, and a few pieces of granulated zinc. The flask is at once connected with the condenser and the contents of the flask are distilled till all ammonia has passed over into the standard acid contained in the precipitating flask previously described and the concentrated solution

can no longer be safely boiled. This operation usually requires from 20 to 40 minutes. The distillate is then titrated with standard ammonia.

The use of mercuric oxide in this operation greatly shortens the time necessary for digestion, which is rarely over an hour and a half in the case of substances most difficult to oxidize and is more commonly less than an hour. In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. Potassium sulphide removes all mercury from solution and so prevents the formation of mercuro-ammonium compounds which are not completely decomposed by soda solution. The addition of zinc gives rise to an evolution of hydrogen and prevents violent bumping. Previous to use the reagents should be tested by a blank experiment with sugar, which will partially reduce any nitrates that are present which might otherwise escape notice.

The following modification must be used for the determination of nitrogen in substances which contain nitrates when it is desired to use this method.

DETERMINATION OF NITROGEN, INCLUDING THE NITROGEN OF NITRATES, BY A MODIFIED METHOD OF KJELDAHL.*

Bring from 0.7 to 1.4 grams of the substance to be analyzed into a Kjeldahl digesting flask, add to this 30.cc of sulphuric acid containing 2 grams of salicylic acid, and shake thoroughly. Then add *gradually* three grams of zinc-dust, shaking the contents of the flask at the same time. Finally, add two or three drops of platinic chloride solution and place the flask on the stand for holding the digestion flasks, where it is heated over a low flame until all danger from frothing has passed. The heat is then raised until the acid boils briskly, and the boiling continued until white fumes no longer pour out of the flask. This requires about five or ten minutes. Add now approximately 0.7 gram mercuric oxide and continue the boiling until the liquid in the flask is colorless, or nearly so. (In case the contents of the flask are likely to become solid before this point is reached add 10 cc. more of sulphuric acid.) Complete the oxidation with a little permanganate of potash in the usual way, and proceed with the distillation as described in the method above.

Determination by the Ruffle Method.

PREPARATION OF REAGENTS.

(1) *A standard solution of sulphuric acid,* half normal, or 19.968 grams SO_3 per liter.

(2) *A standard solution of potassium hydrate,* half normal, or 27.991 grams KOH per liter.

(3) *An alcoholic solution of cochineal.*

(4) *Hyposulphite mixture.*—Prepared by mixing equal parts by weight of soda-lime and finely powdered crystallized sodium hyposulphite.

(5) *Sugar and sulphur mixture.*—This is prepared by mixing equal parts by weight of finely powdered granulated sugar and flowers of sulphur.

(6) *Ordinary granulated soda-lime.*

APPARATUS.

(1) *Combustion tubes* of hard Bohemian glass, 20 inches long and ½ inch internal diameter, drawn to a point.

(2) *Three-bulb, 6-inch U tubes,* with glass stop-cock.

(3) *Aspirator.*

PREPARATION.

(1) Clean and fill the U tube with 10 cubic centimeters of standard acid.

(2) Fit cork and glass connecting tube. Fill the tube as follows: (1) A loosely fitting plug of asbestus, previously ignited, and then 1 to 1½ inches of the hyposul-

* Described by Prof. M. A. Scovell.

phite mixture. (2) The weighed portion of the substance to be analyzed is intimately mixed with from 5 to 10 grams of the sugar and sulphur mixture. (3) Pour on a piece of glazed paper, or porcelain mortar, a sufficient quantity of the hyposulphite mixture to fill about 10 inches of the tube; then add the substance to be analyzed, as previously prepared; mix carefully and pour into the tube; shake down the contents of the tube; rinse off the paper or mortar with a small quantity of the hyposulphite mixture and pour into the tube; then fill up with soda-lime to within 2 inches of the end of the tube. (4) Place another plug of ignited asbestus at the end of the tube, and close with a cork. (5) Hold the tube in a horizontal position, and tap on the table until there is a gas channel all along the top of the tube. Make connection with the U tube containing the acid, aspirate, and see that the apparatus is tight.

The combustion.—Place the prepared combustion tube in the furnace, letting the open end project a little so as not to burn the cork. Commence by heating the soda-lime portion until it is brought to a full red heat. Then turn up slowly jet after jet toward the outer end of the tube, so that the bubbles come off two or three a second. When the whole tube is red hot and the evolution of the gas has ceased and the liquid in the U tube begins to recede towards the furnace, attach the aspirator to the other limb of the U tube, break off the end of the tube, and draw a current of air through for a few minutes. Detach the U tube and wash the contents into a beaker or porcelain basin, add a few drops of the cochineal solution, and titrate.

Determination by the Soda-Lime Method.

Select a tube of hard glass 14 to 16 inches long, draw one end of it to a fine point, and to the other end fit a cork, through which is passed a tube bent at right angles, the other end of which passes through a cork closing tightly one arm of a 6-inch three-bulbed U tube with glass stop-cock. Into the combustion tube first slip a loosely fitting plug of asbestus previously ignited, and then 1½ to 2 inches of soda-lime. Weigh out from 0.7 to 2.8 grams of the substance to be analyzed, and mix it on a piece of glazed paper or porcelain mortar with some finely pulverized soda-lime, and introduce the mixture into the combustion tube. The paper or mortar is then rinsed out with a small quantity of soda-lime and poured into the tube. The tube is then filled up to within 1 to 2 inches of the open end with granulated soda-lime, then with a plug of ignited asbestus. A free passage is formed for the evolved gases by holding the tube in a horizontal position and tapping gently on the table. Introduce the prepared combustion tube into the furnace, letting the open end project a little so as not to burn the cork, supporting the U tube by a clamp. The tube is then gradually heated, commencing at the fore part, nearest the cork, and progressing slowly towards the tail. The combustion should be conducted so as to obtain a steady and uninterrupted flow of gas. When properly carried out the acid in the U tube is never colored. When the acid begins to recede attach the aspirator to the other limb of the U tube, and start it slowly, then break off the point of the combustion tube and draw a current of air through the apparatus for a few minutes, in order to sweep all the ammonia into the acid.

Detach the U tube and wash the contents into a beaker or porcelain basin, add a few drops of an alcoholic solution of cochineal, and titrate.

The standard acid and alkali to be the same as that used in the Ruffle method.

METHOD FOR THE ANALYSIS OF CATTLE FOODS.

Hygroscopic moisture.—Dry 2 to 3 grams at 100° C. to constant weight.

Ash.—Char the substance at a low red heat, exhaust the charred mass with water, burn the insoluble residue, add the ash to the residue from the evaporation of the aqueous extract obtained above, dry the whole at 110°, and weigh. Determine carbonic acid and insoluble matter (sand and charcoal) in the product for the estimation of the pure ash.

Ether extract.—Pulverize the air-dry substance till all of it will pass through a sieve with not less than $\frac{1}{60}$ inch mesh; dry about 5 grams at 100°, and take about 2 grams for each extraction. Exhaust with anhydrous ether of specific gravity .720–.725, not less than eight hours continuously. Dry ether extract at 100° in a current of dry hydrogen to a constant weight.

Crude protein.—Determine nitrogen by the Kjeldahl method in the manner recommended by the Nitrogen Committee, and multiply the result by 6.25 for the crude protein.

Albuminoid nitrogen, Stutzer's method.—Prepare cupric hydrate as follows: Dissolve 100 grams of pure cupric sulphate in 5 liters of water, and add 2.5 cc. of glycerine; add dilute solution of sodium hydrate till the liquid is alkaline, filter, rub the precipitate up with water containing 5 cc. of glycerine per liter, and then wash by decantation or filtration till the washings are no longer alkaline. Then rub the precipitate up again in a mortar with water containing 10 per cent. of glycerine, thus preparing a uniform gelatinous mass that can be measured out with a pipette. Determine the quantity of cupric hydrate per 1 cc. of this mixture.

To 1 gram of the substance, pulverized as for the determination of the ether extract, add 100 cc. of water in a beaker, heat to boiling, or in case of substances rich in starch heat on the water bath ten minutes add a quantity of the cupric hydrate mixture containing 0.7 to 0.8 grams of the hydrate, stir thoroughly, filter when cold, wash with cold water, and without drying put the filter and its contents into the concentrated sulphuric acid for the determination of the nitrogen after Kjeldahl. For filtering use either Schleicher and Shull's No. 589 filter paper or Swedish paper, either of which contains so little nitrogen that it can be left out of account.

If the substance examined consists of seed of any kind or residues of seeds, such as oil cake or anything rich in alkaline phosphate, add a few cubic centimeters of a concentrated solution of alum just before adding the cupric hydrate, and mix well by stirring. This serves to decompose the alkaline phosphate, precipitating aluminum phosphate; if this is not done cupric phosphate and pure alkali may be formed, and the protein copper may be partially dissolved in this alkaline liquid.

Crude fiber, Weende method.—Pulverize as for the ether extract, and extract the fat, at least nearly completely. To 2 grams of the substance in an Erlenmeyer flask add 200 cc. of boiling 1.25 per cent. sulphuric acid, boil immediately and for thirty minutes continuously with as much rotary motion as may be necessary to keep all the substances that may tend to adhere to the sides of the flask in contact with the liquid, throw on a filter of finest linen, and rinse the flask and wash the contents of the filter thoroughly with boiling water. Wash the contents of the filter back into the flask with 200 cc. of a 1.25 per cent. solution of sodium hydrate, at once raise to boiling, and boil continuously for thirty minutes with rotary motion as above described, filter through a Gooch crucible or according to some similar device, and wash very thoroughly with boiling water. Such thorough washing has been found to be essential to success. Wash then with alcohol and finally with ether, dry at 110°, weigh, incinerate, and give the loss of weight for crude fiber.

*Crude fiber closed digestion method.**—Pulverize as for the ether extract, and extract the fat at least nearly completely from 2 grams of substance; allow the ether to evaporate, and transfer to a patent-stoppered beer-bottle with the aid of a funnel and 200 cc. of a solution of 4 per cent. hydrochloric acid in a wash-bottle. Cork, and after seeing that the material is thoroughly saturated with the acid, with none floating on the surface, place in a bath of boiling water of such a size that the neck of the bottle being covered with a copper ring with a suitable-sized hole the entire bottle, except the upper part of the neck, will be surrounded by the boiling water or steam. At intervals of five minutes the bottle is grasped about the neck with a cloth and given such a rotary motion as to stir up the entire contents and wash down the neck. This is preferable to ordinary shaking. The digestion is continued for one hour and the

* Described by Clifford Richardson.

acid then removed by filtration on finest washed linen, as in the preceding method, and the residue washed back with a wash-bottle into the bottle with 200 cc. of 4 per cent. solution of *sodic* hydrate, such as the granulated of the Greenbank Alkali Works. The digestion is then continued as before for one hour and the residue collected, as in the preceding method, and thoroughly washed with hot water, alcohol, and ether. It is dried at 110°, weighed, ignited at as low a heat as possible to prevent volatilization of any soda salts which may not have been removed, and the crude fiber calculated from the loss.

Statement of results.

	In the substance in its natural condition (or as received).	In the dry substance.
Moisture		
Ash		
Ether extract		
Crude fiber		
Crude protein		
Nitrogen: Total		
Albuminoid		

METHODS OF ANALYSES OF DAIRY PRODUCTS.

Butter.

MICROSCOPIC EXAMINATION.

Place a small portion of the fresh sample, taken from the inside of the mass, on a slide, add a drop of pure sweet oil, cover with gentle pressure, and examine with a $\frac{1}{2}$ to $\frac{1}{5}$ inch objective for crystals of lard, &c.

Examine same specimen with polarized light and selenite plate.

Pure fresh butter will neither show crystals nor a parti-colored field with selenite. Other fats, melted and cooled and mixed with butter, will usually present crystals and variegated colors with the selenite plate.

SPECIFIC GRAVITY.

Take a specific gravity flask, not graduated, the stopper of which carries a delicate thermometer, whose bulb occupies sensibly the center of the flask. The thermometer should be first compared with a standard instrument.

Clean the picnometer with water, alcohol, and ether; dry, be careful to remove all the ether vapor, and weigh.

Fill with distilled water, insert stopper, and place in vessel containing water which comes nearly to top of picnometer (without stopper); gradually raise the temperature of water in vessel containing flask until it is 40°.5–41° C.

The moment the thermometer of the picnometer marks 40° remove the flask, dry carefully, cover capillary safety-tube, and set in balance or desiccator to cool. Weigh when temperature falls nearly to that of balance room. Having determined the titre of the flask it is to be carefully dried, filled with the melted and filtered fat at a temperature below 40°, and the weight of the fat determined at 40°, as directed above.

MELTING-POINT.

Apparatus.—The apparatus, Fig. 1, consists of (1) an accurate thermometer, for reading easily tenths of a degree; (2) a less accurate thermometer for measuring the temperature of water in the large beaker glass; (3) a tall beaker glass, 35 cm. high and 10 cm. in diameter; (4) a test tube 30 cm. high and 3.5 cm. in diameter; (5) a stand for supporting the apparatus; (6) some method of stirring the water in the beaker—for example, a blowing bulb of rubber and a bent glass tube extending to near the bottom of the beaker; (7) a mixture of alcohol and water of the same specific gravity as the fat to be examined.

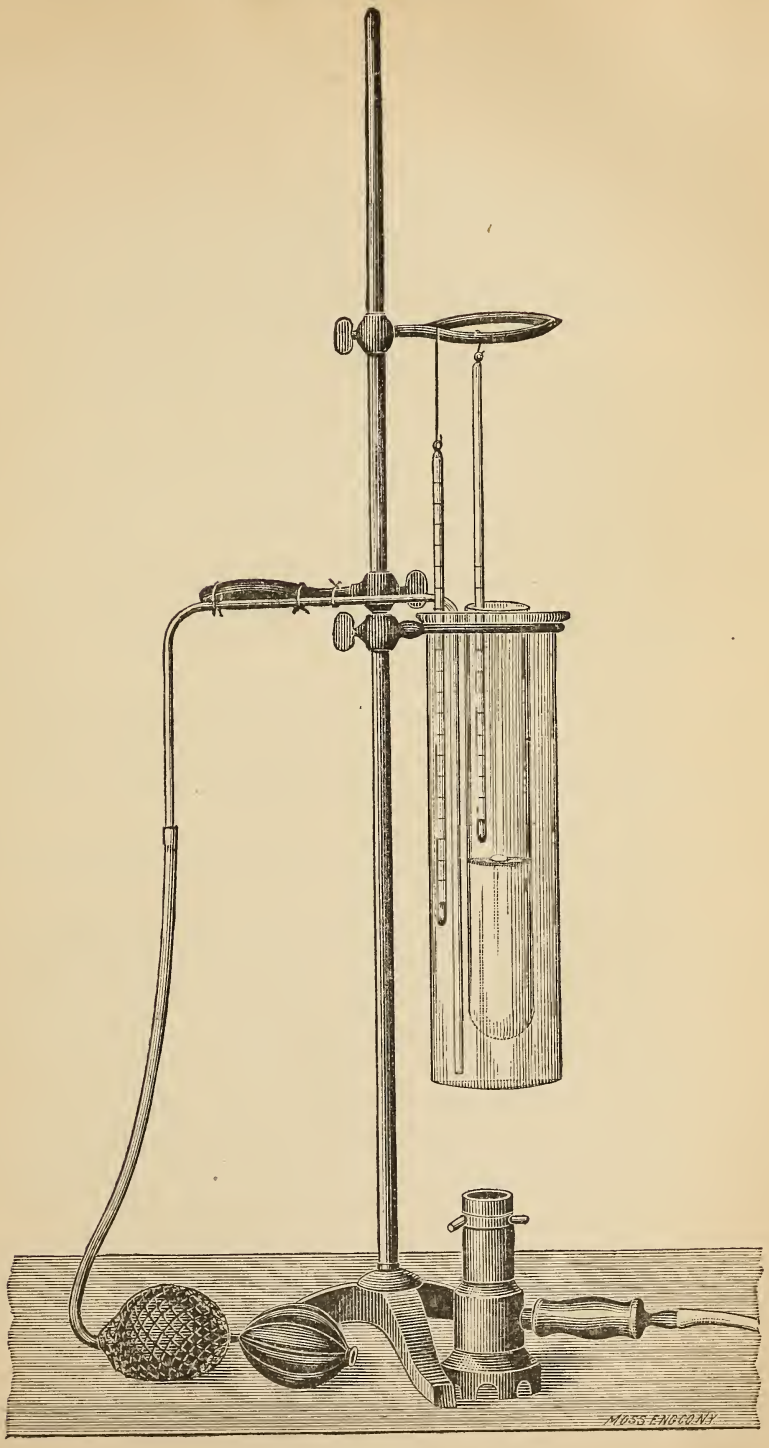

FIG. 1.

Manipulation.—The disks of the fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube from a height of 15 to 20 cm. on to a smooth piece of ice floating in water. The disks thus formed are from 1 to 1½ cm. in diameter and weigh about 200 milligrams. By pressing the ice under the water the disks are made to float on the surface, whence they are easily removed with a steel spatula.

The mixture of alcohol and water is prepared by boiling distilled water and 95 per cent. alcohol for ten minutes to remove the gases which they may hold in solution While still hot the water is poured into the test-tube already described until it is nearly half full. The test-tube is then filled with the hot alcohol. It should be poured in gently down the side of the inclined tube to avoid too much mixing. If the tube is not filled until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on the disk of fat as the temperature rises and finally force it to the top of the mixture.

The test tube containing the alcohol and water is placed in a vessel containing cold water, and the whole cooled to below 10° C. The disk of fat is dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol-water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

The delicate thermometer is placed in the test tube and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk the thermometer is moved from time to time in a circularly, pendulous manner. A tube prepared in this way will be suitable for use for several days; in fact, until the air bubbles begin to attach themselves to the disk of fat. In no case did the two liquids become so thoroughly mixed as to lose the property of holding the disk at a fixed point, even when they were kept for several weeks.

In practice, owing to the absorption of air, it has been found necessary to prepare new solutions every third or fourth day.

The disk having been placed in position, the water in the beaker glass is slowly heated and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6° below the melting-point, the disk of fat begins to shrivel, and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotary movement should be given to the thermometer bulb, which might be done with a kind of clockwork. The rise of temperature should be so regulated that the last 2° of increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so the reading of the thermometer is to be made. As soon as the temperature is taken, the test tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. It is not necessary to cool the water in the bath. The test tube (ice water being used as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum about 1°.5 above the melting-point of the fat under examination.

Working thus with two tubes about three determinations can be made in an hour.

After the test tube has been cooled the globule of fat is removed with a small spoon attached to a wire before another disk of fat is put in.

VOLATILE ACIDS.

Take about 2.5 grams filtered fat in 200 cc. flask, to be used in suos equent distillation; add 2 cc. aqueous solution potassium hydrate containing .5 grams KOH per cc. Add 2 cc. 95 per cent. alcohol and heat on water bath, with occasional agita-

tion. Saponification is complete in a few minutes. Evaporate until alcohol is all driven off, using a current of air to remove alcoholic vapor. The flask is fitted with a delivery tube and condenser, as in Fig. 2. The delivery tube is carried up about 8 inches before it is bent to enter the condenser, and a bulb is blown in it just below the elbow, and this is filled with broken glass or glass wool.

The fatty acids are then set free with 20 cc. of a solution of phosphoric acid made by dissolving 200 grams glacial phosphoric acid in water and making up to one liter. Enough water is added to make the volume of liquid in the flask 70 cc. Distillation is continued until the distillate measures 50 cc., which is titrated with $\frac{N}{10}$ NaOH, using phenol-pthalein as indicator.

(Care should be taken to have the potash used free from carbonate. A potash containing carbonate saponifies a fat with great difficulty. In an instance where the saponification was taking place very slowly, the potash was found to contain 6.80 per cent. CO_2, equal to 21.17 per cent. K_2CO_3.)

SOLUBLE ACIDS.

The washings obtained in the process of estimating the insoluble acids, the description of which follows, are made up to one liter and an aliquot part taken for titration with one-tenth N. alkali, using phenol-phthalein as indicator.

Method for the Determination of Soluble and Insoluble Acids.

REAGENTS.

1. A standard semi-normal hydrochloric-acid solution, accurately prepared.

2. A standard decinormal soda solution, accurately prepared; each 1 cc. contains .0040 grams of NaOH and neutralizes, .0088 grams of butyric acid, $C_4H_8O_2$.

3. An approximately semi-normal alcoholic potash. Dissolve 40 grams of good stick potash in 1 liter of 95 per cent. alcohol, redistilled. The solution must be clear and the KOH free from carbonates.

4. A 1 per cent. solution of phenol-pthalein in 95 per cent. alchol.

Saponification is carried out in rubber-stoppered beer-bottles holding about 250 cc.

About 5 grams of the melted butter fat, filtered and freed from water and salt, are weighed out by means of a small pipette and beaker, which are reweighed after the sample has been taken out and run into a saponification bottle; 50 cc. of the semi-normal potash is added, the bottle closed and placed on the steam bath until the contents are entirely saponified, facilitating the operation by occasional agitation. The alcoholic potash is measured always in the same pipette and uniformity further insured by always allowing it to drain the same length of time, viz, 30 seconds. Two or three blanks are also measured out at the same time and treated in the same way.

In from 5 to 30 minutes, according to the nature of the fat, the liquid will appear perfectly homogeneous, and when this is the case the saponification is complete, and the bottle may be removed and cooled. When sufficiently cool the stopper is removed and the contents of the flask rinsed with a little 95 per cent. alcohol into an Erlenmeyer flask of about 250 cc. capacity, which is placed on the steam bath, together with the blanks, until the alcohol has evaporated.

Titrate the blanks with semi-normal HCl, using phenol-phthalein as an indicator. Then run into each of the flasks containing the fat acids 1 cc. more semi-normal HCl than is required to neutralize the potash in the blanks. The flask is then connected with a condensing tube 3 feet long, made of small glass tubing, and placed on the steam bath until the separated fatty acids form a clear stratum on the surface of the liquid. The flask and contents are then allowed to become thoroughly cold, ice water being used for cooling.

The fatty acids having quite solidified, the contents of the flask are filtered through a dry filter paper into a liter flask, in the manner shown in Fig. 3, care being taken not to

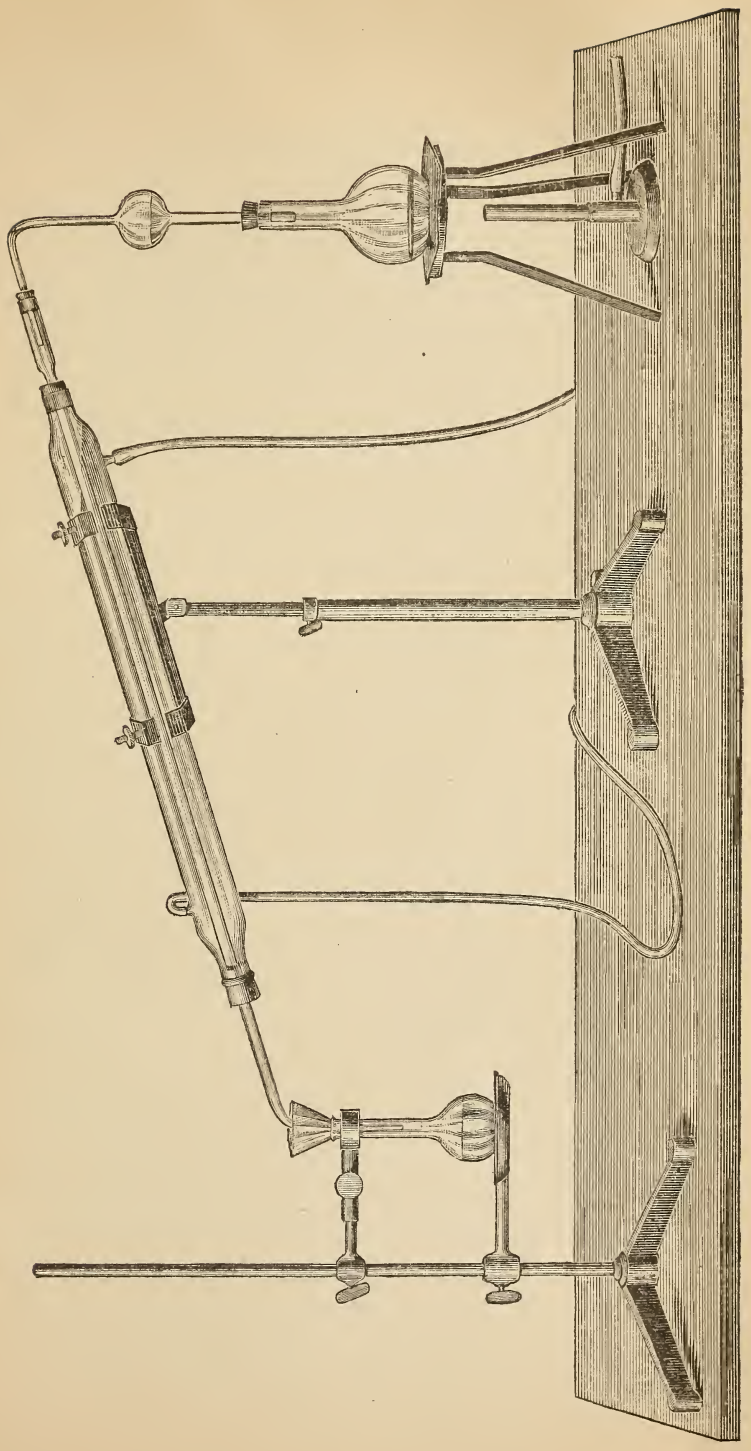

FIG. 2.

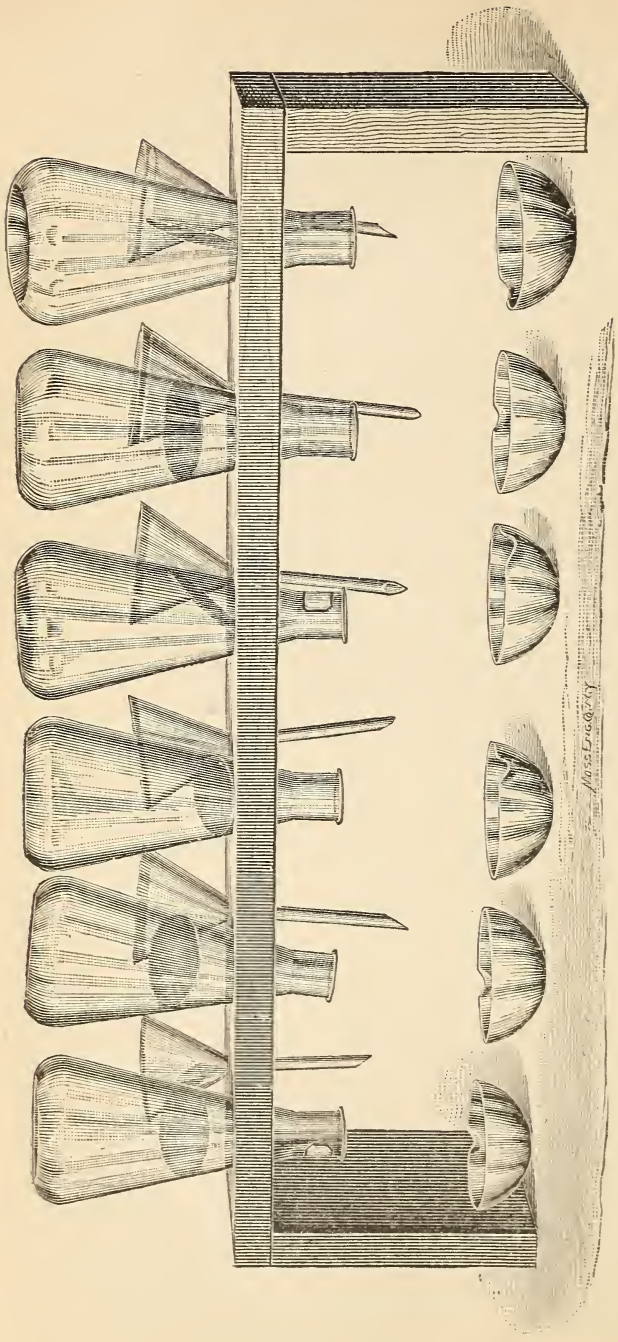

FIG. 3.

break the cake. Two hundred to three hundred cubic centimeters of hot water is next poured on the contents of the flask, the cork with its condenser tube reinserted, and heated on the steam bath until the cake of acids is thoroughly melted, the flask being occasionally agitated with a circular motion, so that none of its contents are brought on the cork. When the fatty acids have again separated as an oily layer, the flask and its contents are cooled in ice water, and the liquid filtered through the same filter into the same liter flask. This treatment with hot water, followed by cooling and filtration of the wash water, is repeated three times, the washings being added to the first filtrate. The mixed washings and filtrate are next made up to 1 liter and 100 cc., in duplicate, is taken and titrated with decinormal NaOH. The volume required is calculated to the whole liquid. The number so obtained represents the measure of decinormal NaOH neutralized by the soluble fatty acids of the butter fat taken, plus that corresponding to the excess of the standard acid used, viz, 1 cc. The amount of soda employed for the neutralization is to be diminished, for the 1 liter, by 5 cc., corresponding to the excess of 1 cc. $\frac{1}{2}$ N. acid.

This corrected volume, multiplied by the factor 0.0088, gives the butyric acid in the weight of butter fat employed. (See table.)

The flask containing the cake of insoluble fat acids is inverted and allowed to drain and dry for twelve hours (Fig. 3), together with the filter paper through which its soluble fatty acids have been filtered. When dry the cake is broken up and transferred to a weighed glass evaporating dish. Remove from the dried filter paper as much of the adhering fat acids as possible and add them to the contents of the dish. The funnel, with the filter paper, is then placed in an Erlenmeyer flask, a hole is made in the bottom of the filter paper, and it is thoroughly washed with absolute alcohol from a wash-bottle. The flask is rinsed with the washings from the filter paper and pure alcohol, and these transferred to the evaporating dish. The dish is placed on the steam bath and the alcohol driven off. It is then transferred to the air bath and dried at 100° C. for two hours, taken out, cooled in a desiccator, and weighed. It is then again placed in the air bath and dried for another two hours, cooled as before, and weighed. If there is no considerable decrease in weight the first weight will do; otherwise, reheat two hours and weigh. This gives the weight of insoluble fat acids in the quantity taken, from which the percentage is easily calculated.

Table for the calculation of soluble fatty acids.

No. cc. KOH sol.	Equiv.	$\frac{N}{10}$ NaOH.	Equiv.	$\frac{N}{10}$ NaOH.	Equiv.	$\frac{N}{10}$ NaOH.	Equiv.
	Grams.		*Grams.*		*Grams.*		*Grams.*
10	.088	+.25	.0902	+.50	.0924	+.75	.0946
11	.0968	25	.0990	50	.1012	75	.1034
12	.1056	25	.1078	50	.1100	75	.1122
13	.1144	25	.1166	50	.1188	75	.1210
14	.1232	25	.1254	50	.1276	75	.1298
15	.1320	25	.1342	50	.1364	75	.1386
16	.1408	25	.1430	50	.1452	75	.1474
17	.1496	25	.1518	50	.1540	75	.1562
18	.1584	25	.1606	50	.1628	75	.1650
19	.1672	25	.1694	50	.1716	75	.1738
20	.1760	25	.1782	50	.1804	75	.1826
21	.1848	25	.1870	50	.1892	75	.1914
22	.1936	25	.1958	50	.1980	75	.2002
23	.2024	25	.2046	50	.2068	75	.2090
24	.2112	25	.2134	50	.2156	75	.2178
25	.2200	25	.2222	50	.2244	75	.2266
26	.2288	25	.2310	50	.2332	75	.2354
27	.2376	25	.2398	50	.2420	75	.2442
28	.2464	25	.2486	50	.2508	75	.2530
29	.2552	25	.2574	50	.2596	75	.2618
30	.2640	25	.2662	50	.2684	75	.2706

The table gives the weight of soluble fatty acids (butyric, &c.) for each quarter of a cubic centimeter of decinormal alkali from 10 to 30.

Example: Weight fat taken ..... grams..	4.967
No. cc. $\frac{N}{10}$ alkali used	25.50
Less 5 cc. due to 1 cc. $\frac{N}{2}$ acid	20.50
Weight soluble fat acids	.1804
Per cent. soluble fat acids	3.63

SAPONIFICATION EQUIVALENT.

About 2.5 grams butter fat (filtered and free from water) are weighed into a patent rubber-stoppered bottle and 25 cc. approximately semi-normal alcoholic potash added. The exact amount taken is determined by weighing a small pipette with the beaker of fat, running the fat into the bottle from the pipette and weighing beaker and pipette again. The alcoholic potash is measured always in the same pipette, and uniformity further insured by always allowing it to drain the same length of time (thirty seconds). The bottle is then placed in the steam bath together with a blank, containing no fat. After saponification is complete, and the bottles cooled, the contents are titrated with accurately semi-normal hydrochloric acid, using phenol-phthalein as an indicator. The number of cubic centimeters of the acid used for the sample deducted from the number required for the blank gives the number of cubic centimeters which combines with the fat, and the saponification equivalent is calculated by the following formula, in which W equals the weight of fat taken in milligrams and N the number of cubic centimeters which has combined with the fat.

$$\text{Sap. Equiv.} = \frac{2\,W}{N}.$$

If it is desirable to express the number of milligrams of potash for each gram of fat employed it can be done by dividing 5,610 by the saponification equivalent and multiplying the quotient by ten.

WATER.

Place about 2 grams in flat-bottomed platinum dish two-thirds full of clean, dry sand and heat for two hours in air bath at 105° C. (For alternate method of estimation of water see page 73.)

ESTIMATION OF SALT.

Volumetric method.—The amount of the butter or butter substitute to be taken is 5–10 grams; weigh in a counterpoised beaker-glass. The butter (fresh from the refrigerator) is placed in portions of about 1 gram at a time in the beaker, these portions being taken from different parts of the sample. By this means a reasonably fair sample of the whole is obtained.

The given quantity having been weighed out, it is removed from the pan.

Hot water is now added (about 20 cc.) to the beaker, continuing the butter, and after it has melted the liquid is poured into the bulb of the separating apparatus (Fig. 4). The stopper is now inserted and the contents shaken for a few moments.

After standing until the fat has all collected on top of the water, the stop-cock is opened and the water, containing most of the salt, is allowed to run into an Erlenmeyer flask, being careful to let none of the fat globules pass.

Hot water is again added to the beaker and thence poured into the separatory apparatus, the bottle well shaken, and the foregoing process is repeated ten to fifteen times, using each time 10–20 cc. water.

The resulting washings contain all but a mere trace of the NaCl, originally present in the butter.

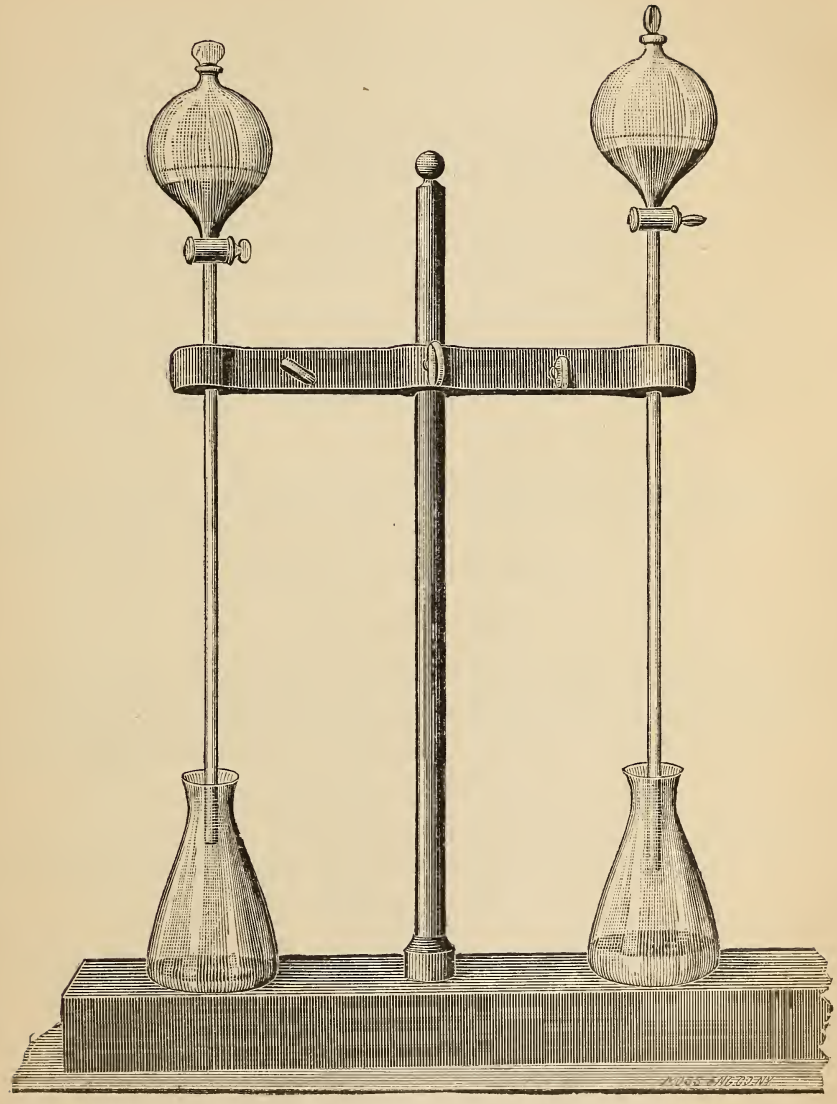

FIG. 4.

Estimation of NaCl in filtrate.—The chloride of sodium is now determined in the filtrate by a standard solution of $AgNO_3$, using a few drops of a saturated solution of potassium chromate as indicator.

TOTAL MINERAL MATTER (ASH) AND CURD.

The methods of estimating curd depend on the principle of first drying a weighed portion of the butter, and afterwards extracting the fat with ether or petroleum. The residual mass is then weighed and the curd determined by loss on ignition. This process is carried on as follows:

Five to ten grams of butter are dried at 100°C., for a few hours in a porcelain dish. The dried fat, &c., is filtered through a Gooch crucible, the contents of the dish all brought into the crucible and well washed with ether or light petroleum. The filter crucible is dried for two hours in air bath and weighed. The curd is then determined by loss of weight on ignition.

The total mineral matter is represented by the residue left after ignition.

Curd and insoluble substances may be estimated somewhat more accurately as follows:

Place the butter (5–10 grams) in a Gooch crucible; set the crucible on a pad of blotting-paper in an air bath (100° C.) for an hour, wash two or three times with ether or light petroleum. Dissolve the salt with hot water, dry at 100° C. and weigh. Any insoluble mineral matter and the ash of the curd is weighed with it in this method.

CASEIN.

Method of Babcock.—Ten grams of the dried butter are treated with light petroleum until all fat is removed. The residue is then ignited with soda-lime or treated by the Kjeldahl method.

METHODS OF MILK ANALYSIS.

ESTIMATION OF WATER.

From a weighing bottle take 5 cc. milk, put in a weighed thin glass dish, or dish lined with tinfoil, one-third full of powdered asbestos; dry for two hours at 100° C. The temperature obtained in a boiling-water bath does not reach 100° C. The milk should be dried in an air bath, the temperature of which is carefully controlled.

ALTERNATE METHOD OF ESTIMATING WATER.

Evaporate one to two grams of milk in shallow watch glass or platinum dish on he water bath for thirty minutes. Dry for an hour at 100° C. and weigh.

ESTIMATION OF CASEIN.

Take 5 grams of milk, digest in Kjeldahl apparatus, with 20 cc. H_2SO_4, and estimate ammonia in the usual way.

ALTERNATE METHOD OF ESTIMATION OF CASEIN.

Rub up in a mortar the thin dish containing the dried residue from the above process or remove the foil containing it, and transfer to soda-lime combustion tube in the usual way.

The mortar and pestle must be well cleaned with the soda-lime and these cleanings placed in the tube.

Or the dish or tinfoil and its contents may be transferred to a digestion flask and the casein estimated by the method of Kjeldahl.

ESTIMATION OF FAT.

Method of Adams.—The kind of paper and the method of using it first proposed by Adams are as follows:

"As for material, the only extra article is some stout white blotting-paper, known in the trade as 'white demy blotting mill 428,' weighing 38 pounds per ream. This should be in unfolded sheets, machine-cut into strips 2¼ inches wide and 22 inches long; each sheet in this manner cuts into seven strips.

"I have tried other papers, but none have answered so well as this; it is very porous and just thick enough. Each of these strips is carefully rolled into a helical coil, for which purpose I use a little machine, made by myself, consisting of a stout double wire, cranked twice at right angles, and mounted in a simple frame. One end of the strip being thrust between the two wires, the handle is turned, and the coil made with great facility. This may be done, for the nonce, on a glass rod, the size of a cedar pencil. Two points have to be carefully attended to: the paper must not be broken, and the coil must be somewhat loose, the finished diameter being a little under an inch. I am in the habit of rolling up a considerable number at a time and placing each within a brass ring as it is rolled, inscribing on one corner with a lead-pencil its own proper number.

"These coils are next thoroughly dried, and I need hardly say the accuracy of the process depends upon this drying. This can be satisfactorily done in an ordinary air bath at 100° C., providing the bath be heated properly and the paper kept in it long enough. I found the common way of heating the thin bottom of the bath with a single jet not to answer. My bath is placed upon a stout iron surface, which is heated by a large ring of jets; in this way the heat is evenly distributed over the whole of the bottom of the bath, and the papers, which are put in a cage frame of tinned iron wire 5 by 2½ inches and divided into eight partitions, get evenly and completely dried, if allowed to remain in the bath all night, and weighed in a weighing tube next morning, and their weights having been registered according to their numbers, stored away ready for use, as follows:

"The milk to be examined is shaken, and with a pipette 5 cc. are discharged into a small beaker 2 inches high by 1¼ diameter, of a capacity of about 30 cc. weighing about 12 grams. This charged beaker is first weighed, and then a paper coil gently thrust into the milk very nearly to the bottom. In a few minutes the paper sucks up nearly the whole of the milk. The paper is then carefully withdrawn by the dry extremity of the coil and gently reversed, and stood, dry end downwards, on a clean sheet of glass. With a little dexterity all but the last fraction of a drop can be removed from the beaker and got on the paper. The beaker is again weighed, and the milk taken got by difference. It is of importance to take up the whole of the milk from the beaker, as I am disposed to consider the paper has a selective action, removing the watery constituents of the milk by preference over the fat.

"The charged paper is next placed in the water oven on the glass plate milk-end upwards, and rough-dried. Mismanagement may possibly cause a drop to pass down through the coil onto the glass. This accident ought never to occur; but if it does, it is revealed in a moment by inspection of the surface of the glass, and the experiment is thereby lost.

"In about an hour it is rough-dried and in a suitable condition for the extraction of the fat."

The method of Adams has been thoroughly tried by the English chemists, and has received the approval of the English Society of Public Analysts. It gives uniformly about .2 per cent. more fat in normal milk than the ordinary gravimetric methods.

The following modification of the process may be used:

"The blotting-paper is replaced by thick filtering paper cut into strips 2 feet long and 2.5 inches wide. These are thoroughly extracted by ether or petroleum ether (or alcohol).

"One end of the strip of paper being held horizontally by a clamp or by an assistant, 5 cc. milk is run out by a pipette from a weighing bottle along the middle of the strip of filtering paper, being careful not to let the milk get too near the ends of the paper, and to secure an even distribution of it over the whole length of the slip. The pipette is replaced in the weighing bottle and the whole reweighed, and thus the quantity of milk taken is accurately determined. The strip of paper is now hung up over a sand bath in an inclosed space high enough to receive it where the air has a temperature of 100° C. (*circa*). In two or three minutes the paper is thoroughly dry. It is at once, while still hot, rolled into a coil and placed, before cooling, in the extraction apparatus already described.

"The fat is dissolved by ether or petroleum, collected in a weighed flask, and, after thorough drying, weighed."

The fat after extraction may also be estimated volumetrically, as described in the method of Morse.

From data which have been collected it appears that the estimation of the fat in milk by the lactocrite is strictly comparable with the results of the Adams method. Those who have this instrument, therefore, can use it instead of the method given.

ALTERNATE METHOD OF ESTIMATING THE FAT IN MILK.

Method of Morse, Piggot, and Burton.—This method consists in the dehydration of the milk by means of anhydrous sulphate of copper; the extraction of the fat by means of the low-boiling products of petroleum; the saponification of the butter by means of an excess of a standard solution of potassium hydroxide in alcohol; and the determination of the excess of the alkali by means of a solution of hydrochloric acid. The following apparatus and reagents are required:

(1) A porcelain mortar and pestle.

(2) An extraction tube, 14 or 15 mm. in diameter, 220 mm. in length, with funnel-shaped top. A straight chloride of calcium tube may be used for this.

(3) A 200 cc. Erlenmeyer flask, strong enough to be used with a filter pump.

(4) A suitable stand for holding the flask and extraction tube.

(5) Ten-cubic centimeter pipettes.

(6) Weighing-glasses with ground-glass stoppers.

(7) A low-boiling gasoline, distilling between 30° and 60°.

(8) Dehydrated sulphate of copper.

(9) Semi-normal solution of potash in 95 per cent. alcohol.

(10) A semi-normal solution of hydrochloric acid.

Manipulation.—Place about 20 grams of the anhydrous copper sulphate, roughly measured in a copper spoon of the size to hold about that amount, in a porcelain mortar; make a cavity in the center of the mass with the pestle. Allow 10 cc. of the milk to run on to the copper sulphate, being careful that none of it touches the sides of the mortar. When the milk is nearly dry, grind the mass up with a little clean sand, transfer to the extraction tube, gently pressing it down in the tube by means of a glass rod. The lower portion of the extraction tube to be packed with clean cotton wool. The fat is extracted in the following way: 15 cc. of benzine are poured over the material in the extraction tube and drawn down with the aid of the filter pump, until the whole of the mass to be extracted has become wet with the liquid, when the connection with the pump is closed; after about five minutes another portion of 15 cc. of benzine is poured into the tube and the whole of the liquid slowly drawn through with aid of the pump into the flask. Usually one extraction of this kind is sufficient to withdraw the whole of the butter, but for the sake of greater accuracy the process may be repeated two or three times.

Titration.—The benzine may be evaporated and the residual butter fat saponified with about 25 cc. of the approximately semi-normal potash. The residual alkali is determined by means of the semi-normal hydrochloric acid, using phenol-phthalein as indicator. The difference between the amount required in this process and the amount necessary to neutralize the quantity of alkali taken gives the amount of alkali required for the saponification. The number of milligrams of potash required for one gram of the fat is taken at 230. The fat may also be accurately titrated without evaporating the benzine.

ALTERNATE METHOD OF ESTIMATING WATER AND FAT IN MILK.

Method of Babcock.—In the bottom of a perforated test tube is placed a clump of clean cotton; the tube is then filled three-quarters full of ignited asbestus, lightly packed, and a plug of cotton inserted over it. The tube and contents are weighed and the plug of cotton carefully removed and 5 grams of milk from a weighed pipette run into it, and the plug of cotton replaced. The tube connected at its lower end by a rubber tube and adapter with a filter pump is placed in a drying oven of a 100° C., and a slow current of dry air drawn through it until the water is completely expelled which in no case requires more than two hours.

The tube containing the solids from the above operation is placed in an extraction apparatus and exhausted with ether in the usual way.

ALTERNATE METHOD OF ESTIMATING WATER AND FAT.

Method of Professor Macfarlane.—A glass tube 4–5 cc. in length and 2 cm. in diameter, open at one end, drawn out to a tube 5 mm. in diameter at the other end, is two-thirds filled with asbestus fiber, such as is used in manufacturing packing. It is dried in the water bath for several hours, cooled in the desiccator, and weighed. Ten cubic centimeters of the milk is then added from a pipette, which is completely absorbed by the asbestus. It is then weighed, the additional weight of the milk representing the amount taken. The tube, along with many others, is placed in a water bath with constant level and dried for ten or twelve hours (during the night) at a temperature of 90°. Next morning the tubes are cooled in the desiccator and weighed, the loss in weight being the moisture. The tubes are then placed in the Soxhlet extraction apparatus and exhausted with petroleum ether for four hours. They are then removed and dried in a steam bath, cooled in desiccator, and weighed. The loss represents the butter fat.

THE ESTIMATION OF SUGAR.

The reagents, apparatus, and manipulation necessary to give the most reliable results in milk sugar estimation are as follows:

Reagents.—(1) *Basic plumbic acetate*, specific gravity 1.97. Boil a saturated solution of sugar of lead with an excess of litharge, and make it of the strength indicated above. One cubic centimeter of this will precipitate the albumens in 50 to 60 cc. of milk.

(2) *Acid mercuric nitrate.* Dissolve mercury in double its weight of nitric acid, specific gravity 1.42. Add to the solution an equal volume of water. One cubic centimeter of this reagent is sufficient for the quantity of milk mentioned above. Larger quantities can be used without affecting the results of polarization.

(3) *Mercuric iodide with acetic acid.* KI 33.2 grams, $HgCl_2$ 13.5 grams, H, C_2H_3O 20 cc., H_2O 64 cc.

Apparatus.—(1) Pipettes marked at 59.5, 60, and 60.5 cc. (2) Sugar flasks marked at 102.4 cc. (3) Filters, observation tubes, and polariscope. (4) Specific gravity spindle and cylinder. (5) Thermometers.

Manipulation.—(1) The room and milk should be kept at a constant temperature. It is not important that the temperature should be any given degree. The work can be carried on equally well at 15° C., 20° C., or 25° C. The slight variations in

rotatory power within the above limits will not affect the result for analytical purposes. The temperature selected should be the one which is most easily kept constant.

(2) The specific gravity of the milk is determined. For general work this is done by a delicate specific gravity spindle. Where greater accuracy is required use specific gravity flask.

(3) If the specific gravity be 1.026, or nearly so, measure out 60.5 cc. into the sugar flask. Add 1 cc. of mercuric nitrate solution or 30 cc. mercuric iodide solution and fill to 100.4 cc. mark. The precipitated albumen occupies a volume of about 2.44 cc. Hence the milk solution is really 100 cc. If the specific gravity is 1.030 use 60 cc. of milk. If specific gravity is 1.034 use 59.5 cc. of milk.

(4) Fill up to mark in 102.4 cc. flask, shake well, filter, and polarize.

NOTES.—In the above method of analysis the specific rotatory power of milk sugar is taken at 52.5, and the weight of it in 100 cc. solution to read 100 degrees in the cane sugar scale at 20.56 grams. This is for instruments requiring 16.19 grams sucrose to produce a rotation of 100 sugar degrees. It will be easy to calculate the number for milk sugar whatever instrument is employed.

Since the quality of milk taken is three times 20.56 grams, the polariscopic readings divided by 3 give at once the percentage of milk sugar when a 200 mm. tube is used.

If a 400 mm. tube is employed, divide reading by 6; if a 500 mm. tube is used, divide by 7.5.

Since it requires but little more time, it is advisable to make the analysis in duplicate, and take four readings for each tube. By following this method gross errors or observation are detected and avoided.

By using a flask graduated at 102.4 for 60 cc. no correction for volume of precipitated caseine need be made. In no case is it necessary to heat the sample before polarizing.

In the above method no account is taken of the fat which is retained on the filter with the caseine. It is worth while to inquire if a correction similar to that made for the albuminoids should not also be made for the fat?

ESTIMATION OF ASH.

Evaporate to dryness in a weighed platinum dish 20 cc. of milk from a weighing bottle, to which 6 cc. of HNO_3 has been added, and burn in muffle at low red heat until ash is free from carbon.

Appendix B.

OFFICERS AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS OF THE UNITED STATES FOR 1887-1888.

PRESIDENT,

P. E. CHAZAL, State Chemist of South Carolina.

VICE-PRESIDENT,

W. J. GASCOYNE, State Chemist of Virginia.

SECRETARY,

CLIFFORD RICHARDSON, Chemist to the District of Columbia and Honorary Assistant Chemist U. S. Department of Agriculture.

EXECUTIVE COMMITTEE,

E. H. JENKINS, Connecticut Agricultural Experiment Station.

J. A. MYERS, State Chemist of Mississippi.

COMMITTEE ON PHOSPHORIC ACID.

W. J. Gascoyne, of Virginia.
N. W. Lord, of Ohio.
W. E. Moses, of Tennessee.

COMMITTEE ON POTASH.

John A. Myers, of Missouri.
Wm. Frear, of Pennsylvania.
E. H. Jenkins, of Connecticut.

COMMITTEE ON NITROGEN.

M. A. Scovell, of Kentucky.
N. T. Lupton, of Alabama.
Wm. McMurtrie, of Illinois.

COMMITTEE ON CATTLE FOODS.

G. C. Caldwell, of New York.
W. H. Jordan, of Maine.
Clifford Richardson, of District of Columbia.

COMMITTEE ON DAIRY PRODUCTS.

H. W. Wiley, of District of Columbia.
S. M. Babcock, of New York.
H. P. Armsby, of Pennsylvania.

COMMITTEE ON FERMENTED LIQUORS.

W. B. Rising, of California.
C. A. Crampton, of District of Columbia.
G. F. Fellows, of Maryland.

COMMITTEE ON SUGAR.

W. C. Stubbs, of Louisiana.
N. T. Lupton, of Alabama.
H. W. Wiley, of District of Columbia.

Appendix C.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This Association shall be known as the Association of Official Agricultural Chemists in the United States. The objects shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statements of analysis of fertilizers, soils, cattle-foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the U. S. Department of Agriculture, or with any State or national agricultural experiment station, or agricultural college or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership, and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to a vote in the Association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the Association by presenting proper credentials and signing this constitution. All analytical chemists and others interested in the objects of the Association may attend its meetings and take part in its discussions, but shall have no vote in the Association.

(3) The officers of the Association shall consist of a president, vice-president, and secretary, who shall also officiate as treasurer; and these officers, together with two other members to be elected by the Association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board, whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee to continue in office till the annual meeting next following.

(4) There shall be appointed by the chair, at the regular annual meeting, standing committees of three members each, to be called, respectively, the Committee on Phosphoric acid, the Committee on Potash, the Committee on Nitrogen, the Committee on Cattle-foods, and the Committee on Dairy Products, whose reports shall be taken up, discussed, and disposed of in the order named. Additional committees may be appointed from time to time as the Association may direct. It shall be the duty of these committees to test methods, to distribute samples to members of the Association and others who may signify their desire to participate in the work, and to present a written report at each annual meeting embodying the progress made during the year in analytical methods bearing on their respective topics, and the results of work done under their direction.

(5) The special duties of the officers of the Association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this Association shall be held at such place as shall be decided by the Association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(8) The executive committee shall confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the Association.

(9) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by a vote of two-thirds of the members present and entitled to vote.